

**CHARACTERIZATION OF NMR DIFFUSION
COUPLING PHENOMENA IN CARBONATE
FORMATIONS UNDER THE PRESENCE OF MULTIPLE
RESERVOIR FLUIDS**

BY

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2023

Dedication

To my parents and role models, who have always believed in me and supported
me in everything I do.

To my wife and soulmate. To my son, who is my greatest joy.

To my brothers.

To my family, my true source of inspiration,

I dedicate this thesis to all of you.

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LIST OF ABBREVIATIONS

NMR	:	Nuclear magnetic resonance
T₁	:	Spin-lattice relaxation time
T₂	:	Spin-spin relaxation time
S_r	:	Effective surface
S_{wir}	:	Irreducible water saturation
S_{wi}	:	Water saturation
S_o	:	Oil saturation
CT	:	Computerized Tomography
MICP	:	Mercury Injection Capillary Pressure
ρ₂	:	Surface relaxivity
S/V	:	Surface to volume ratio
D₀	:	Self-diffusion coefficient of the fluid
G	:	Magnetic field gradient
γ	:	Gyromagnetic
CCA	:	Conventional core analysis
SCAL	:	Special core analysis

PFG	:	Pulsed field gradient
t_e	:	Time between NMR spin echoes
IRSE	:	Inversion recovery spin eco
IR	:	Inversion recovery pulse sequence
CPGM	:	Carr-Purcell-Meiboom-Gill sequence

|

ABSTRACT

Full Name : [Fawaz Mohammad Hussain Al-Boghail]
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Carbonate Formations under The Presence of Multiple Reservoir
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NMR have introduced unique, yet revolutionary capabilities that enabled an improved formation evaluation. NMR stands as one of the paramount techniques used to characterize petrophysics formation properties, enabled by applications extending beyond the lab to field measurements. One of the most important evaluations NMR offers is the characterization of pore size distributions which can be used to further understand the pore structure and devise the production strategies to target the hydrocarbon bearing zones accordingly. NMR measurements reflect the time of fluids occupying the pore spaces take to relax after being subjected to magnetization, which is known as spin-lattice relaxation time (T_1) and spin-spin relaxation time (T_2).

However, NMR T_2 relaxations interpretation practices commonly assume that it is solely proportional to surface relaxation which infers pore size distributions, where relaxation of different pore bodies is independent of the others due to disconnection between the bodies. However, different studies have reported an anomalous NMR response in some connected bimodal porosity systems, in what is referred to as “diffusion coupling phenomena”. Owing to the connectivity between multiple porosity systems, and instead of relaxation time reflecting multiple pore bodies, a coupled NMR relaxation time is exhibited. The coupled

relaxation time can mislead to interpret a single pore system of lower quality, reflecting inaccurate formation movable fluids. All published evaluations of diffusion coupling were done on single-phase systems. There is an existing need to evaluate the phenomena under the presence of multiple fluid systems.

This thesis aims to Investigate the effect of NMR fluid properties (diffusion coefficient & bulk relaxations) on diffusion coupling on both controlled medium (synthesized gel & simulation) and core plug samples. Additionally, to evaluate diffusion coupling under the presence of multiple reservoir fluids.

The study revealed that pore geometry effect was consistent with literature findings. Lower contrast between the two pore bodies resulted in stronger diffusion coupling. Both NMR fluid properties, bulk relaxation and diffusion coefficient, have significant impact on diffusion coupling. Those two parameters are interconnected, where each fluid has specific values of bulk relaxation and diffusion coefficient. When evaluated independently through NMR simulator, diffusion coefficient was positively correlated with coupling. Faster bulk relaxation reduces the strength of diffusion coupling. Additionally, the study revealed that diffusion coupling strength in single-phase saturation might be completely different than in multi-phase, owing to the significance of wettability effect.

ملخص الرسالة

الاسم الكامل: فواز محمد حسين البغيل

عنوان الرسالة: توصيف و تحليل ظاهرة الانتشار المقارن في مجال الرنين النووي المغناطيسي في الأوساط المسامية الثنائية في ظل وجود عدة موانع

التخصص: هندسة البترول

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قدمت تقنية الرنين المغناطيسي النووي قدرات فريدة و ثورية مكنت من تحسين دراسة و تقييم التكوينات الجولوجية للمكامن النفطية. و تعد اليوم أحد أهم التقنيات المستخدمة لتوصيف الخصائص البتروفيزيائية من خلال تطبيقاتها التي تمتد من المختبرات إلى القياسات الميدانية. أحد أهم التقييمات التي تقدمها التقنية هي تقدير حجم و توزيع المسامات والتي تستخدم لبناء أفضل استراتيجية ممكنة لانتاج المكامن النفطية .

تعكس قياسات الرنين المغناطيسي النووي الوقت الذي يسغرقه الموانع للاسترخاء بعد تعرضه لحقل من المغنطة. ومع ذلك ، فإن ممارسات تحليل إشارات الاسترخاء تفترض عادة أنها تتناسب فقط مع توزيعات و أحجام المسام، حيث يسترخي كل وسط مسامي بشكل مستقل عن الوسط الآخر. أشارت بعض الدراسات عن استجابة غير طبيعية للرنين المغناطيسي النووي في بعض الأوساط المسامية ثنائية النسق المتصل ، و أعزت السبب في ذلك لظاهرة " الاقتران الانتشاري"، وهو محور هذه الرسالة. تحدث هذه الظاهرة عندما ينتشر جزيء المانع من وسط مسامي إلى آخر، يمكنه من تبادل المغنطة مع جزيئات السائل في المسام الجديد. يمكن أن يسبب هذا التبادل في استرخاء الأجسام بشكل متصل، منتجة لاسترخاء لا يعكس حقيقة الوسط المسامية، إنما يعكس متوسط حجم الوسطين المساميين. يؤدي ذلك إلى فهم غير دقيق للطبيعة البتروفيزيائية للممكن .

تبحث هذه الرسالة ظاهرة الانتشار المقارن في الأوساط ذات المسامات المتعددة، والتي تنشط عادة في المكامن الجيرية، وتركز هذه الدراسة على درجة تأثير خصائص الموانع على هذه الظاهرة. خلصت هذه الدراسة على التأثير المباشر لخصائص الموانع، كثابت الانتشار و استرخاء المانع الحر على حدوث ودرجة حدة هذه الظاهرة.

CHAPTER 1

INTRODUCTION

1.1 Problem Statement

The global energy market has witnessed disruptions in the supply demand cycles due to political climate contentions, outbreak of COVID-19 and lack of investment in the development of oil and gas fields. The issue in financing the development of fields has resulted in the failure of many producing countries to meet their OPEC + assigned targets. This poses a daunting threat to the stability of the future energy market. In response, the kingdom of Saudi Arabia has announced an ambitious plans to increase its maximum sustainable capacity from 12 to 13 million barrels per day, by 2027. (Saba & El Dahan, 2022). Sustaining the current production capacity would require huge investments and extensive engineering efforts. By extension, availing the extra million barrels per day above the current capacity necessitates the absolute best in-class in engineering solutions and field management, including excellence in reservoir characterization to unlock the full potential of fields. NMR would play pivotal role in improving field assessment and reservoir characterization.

NMR stands as one of the paramount techniques used to characterize petrophysics formation properties with application capabilities extending beyond the lab to the field. For

instance, NMR is used to evaluate both pore size and pore throat distributions which can be used to understand the pore structure and estimate permeability. However, NMR T_2 interpretation practices commonly assume that it is solely proportional to pore size distributions, where relaxation of different pore bodies is independent of the others due to disconnection between the bodies. However, different studies have reported an anomalous NMR response in some connected bimodal porosity systems, in what is referred to as “diffusion coupling phenomena”. Owing to the connectivity between multiple porosity systems, and instead of relaxation time reflecting multiple pore bodies, an average NMR relaxation time is exhibited. The pseudo coupled relaxation time can mislead to characterizing a single pore system of lower quality.

Diffusion coupling is a product of multiple factors that pertain to relaxation media, present fluids, and the way they interact with each other. Figure 1 provides an overview of factors affecting NMR diffusion coupling. The relaxation media have a significant effect on diffusion coupling. This is encompassed by grain surface relaxivity, which relates to the composition of the grain’s surface, and pore geometry, which is characterized by the pore system, pore throat and tortuosity. Multiple studies have examined those two factors experimentally and using simulation, confirming the impact of pore geometry and surface relaxivity on the strength of diffusion coupling. On the fluid side, fluids’ unique NMR properties, including fluid diffusion coefficient and bulk relaxation behavior, also impact diffusion coupling, which are the focus of this study. As those properties differ from fluid to another, fluid saturation is considered another factor affecting diffusion coupling. Wettability is at the intersection of those factor, constituting the fifth factor. Those five element collectively affect the phenomena, and some (such as fluid saturation and

wettability) are dependent on other factors. Few studies provided a shallow examination of the effect of fluid diffusion coefficient, considering only single phase. To the author's best knowledge, no paper evaluated the effect of fluid saturations or bulk relaxations on diffusion coefficient. Additionally, no papers in literature examined the phenomena on actual core plug samples.

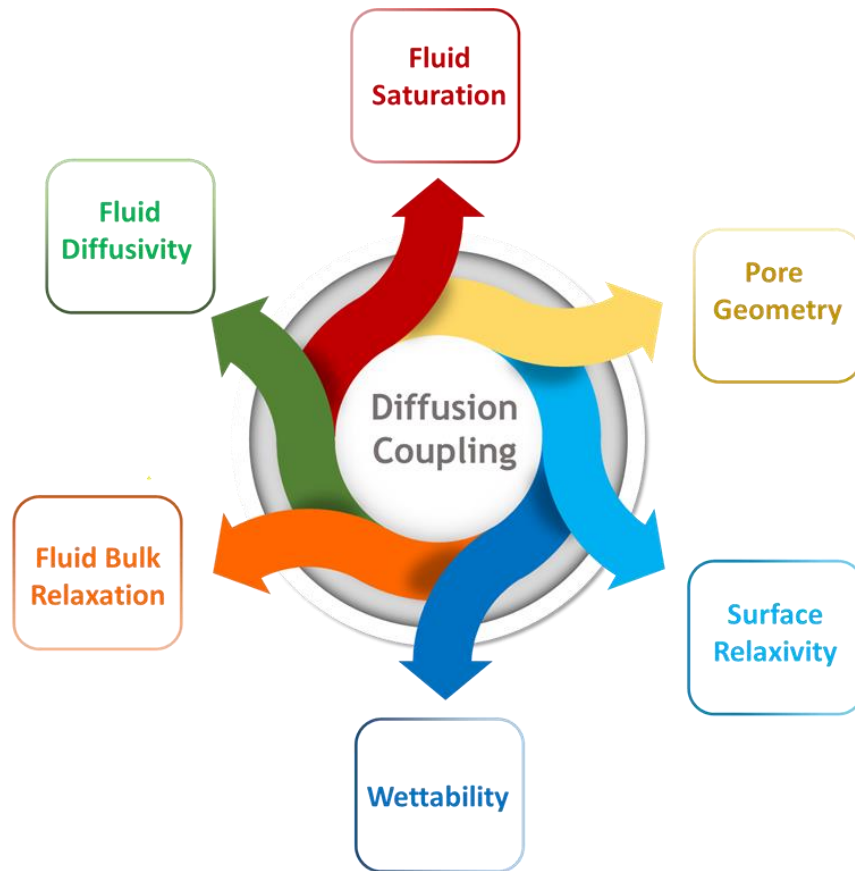


Figure 1: Factors contributing to diffusion coupling phenomena

1.2 Objectives

The objectives of this work are to investigate the diffusion coupling phenomena in bimodal pore systems, with a focus on investigating the role of fluid's NMR properties (diffusion coefficient and bulk free relaxation), and to cross-validate findings with actual core samples. Additionally, to evaluate diffusion coupling under the presence of multiple reservoir fluids. |

| CHAPTER 2 |

| LITERATURE REVIEW

2.1 Computerized and micro computerized tomography

The rapidly growing interest in digital transformations in the oil and gas industry has created a wave of advancements in different frontiers. Computerized Tomography (i.e. CT) and micro-CT scanning techniques are considered to be key transformation enablers, which have been widely used in the industry with expanding portfolio of applications. This section provides an overview of applications of CT scan in petrophysical evaluations.

2.1.1 Overview of CT and micro-CT scan

CT scanning utilizations and applications in the oil and gas industry have been growing, with the advancement in technology, enabling improved resolution. In 1972, CT scanning techniques were adapted in the field of medicine to diagnose internal damages through non-destructive X-ray imaging processes. Multiple localized slices are imaged through X-ray then reproduced through complex mathematical models. (Sarker & Siddiqui, 2009). Building on the effectiveness of CT-Scan, the technique adaptation has spread across many disciplines, including petroleum engineering in the 80s.

The process of CT and Micro-CT in the context of petroleum engineering starts with sample preparation. The formation core samples are plugged into smaller pieces to ease the transportation and analysis of the sample. The analysis of bigger core samples, also known as whole core, is also possible but not as frequent as the smaller plug core samples. After cleaning and fluid extraction, measurements of conventional core analysis are usually conducted as most core analyses require the sample to be in a saturated status. Based on the objective of the test, the sample is saturated with reservoir fluid, mounted to the CT-scan core holder after the equipment has been properly calibrated. After that, based on its length, the sample is digitally dissected into multiple slices to plan for the localized section of image acquisition. Accordingly, CT images are scanned. Through available commercial software which uses complex mathematical algorithms, the images are processed to reconstruct the model of the sample, ultimately producing the objective petrophysical data. Figure 2 and

Figure 3 provide an overview of the CT scanning processes. Details of workflows and processes of measurements done using CT-scan and micro-CT-scan can be found in (Saxena et al, 2017), (Song et al, 2019) and (Elkhoury et al, 2019). The difference in resolution between the micro and the conventional medical CTs lies in their resolution capabilities. The conventional features a resolution in the scale of millimeter (0.5 – 3); whereas the micro-CT resolution is in the micrometer scale (1 – 30). This translates to data output gridding of 512 x 512 for the conventional CT and up to 2048 x 2048 for the micro-CT.

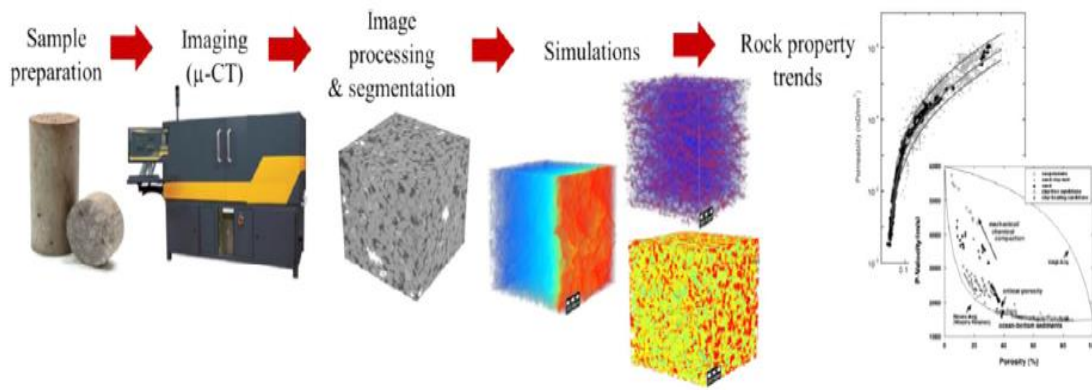


Figure 2: Overview of the process of formation core plug scanning via micro-CT scan (Saxena 2017).

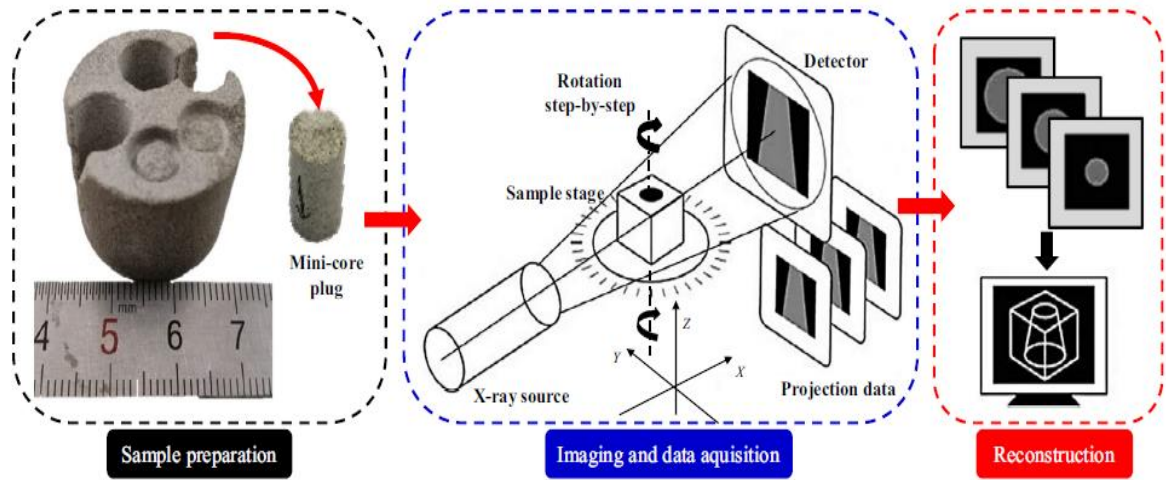


Figure 3: Overview of processes followed to image a plug sample via micro-CT scan (Song, 2019)

2.1.2 Applications and Limitations

Due to the effectiveness of CT-Scanning techniques in visualizing fine-scale mediums, the technique applications have witnessed exponential and unprecedented growth in the field of petroleum engineering and petrophysics. In the context of core analysis, CT-scanning applications were used for two main functions. The first is to provide a petrophysical characterization of the core. The second is to characterize the rock-fluids interactions under flowing conditions. (Sarker & Siddiqui, 2009).

The most widely used application of CT scanning is pore network characterization and modeling, which is a mean of developing a localized geo-model under static conditions. Early applications of CT scanning were inspired by the medical applications, which focuses primarily on tomography. That was the trigger for tomography legacy applications on core analysis in the 1980s, which laid the foundations for the expansion of CT-scan applications.

Resemblance of early tomography applications to medical industry can be observed in (Hunt & Englur, 1988). Figure 4 provide examples of CT tomography characterization, while Figure 5 demonstrate examples of CT scan pore size distribution assessment. Currently, CT scanning can provide valuable core characteristics such as pore size and network, pore throat, cross section visualization, density distributions, fracture, and micro fracture detections. According to Withjack et al, Whole-core CT scanning analysis were used as mean of quality assurance to well-log correlations. (2003). Pore size modeling can provide valuable insight into the pore connectivity and size distributions, which can be of significant importance when supplemented with other techniques such as NMR or MICP for improved rock characterization and analysis. Figure 7 provide an example of three-dimensional reconstructed pore models. Another valuable application of CT scan is the pore throat distribution and modeling, which is of equal importance and is complementary to pore modeling, examples of pore size and throat modeling are presented in Figure 8 and Figure 9.

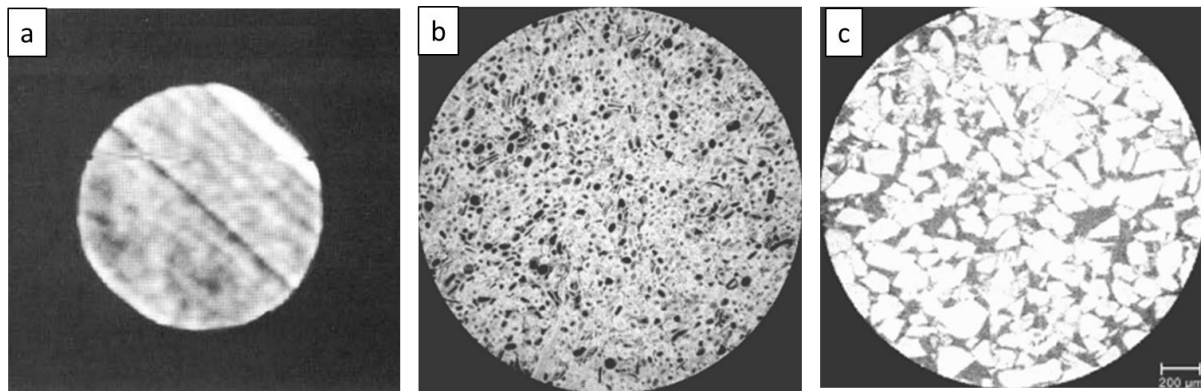


Figure 4: Advancement in tomography characterization. a) Homogenous Berea core (Hunt, 1988). b) Carbonate sample characterized by conventional CT (Withjack et al, 2003). c) Cross section of unconsolidated sand characterized by micro-CT (Song et al, 2019).

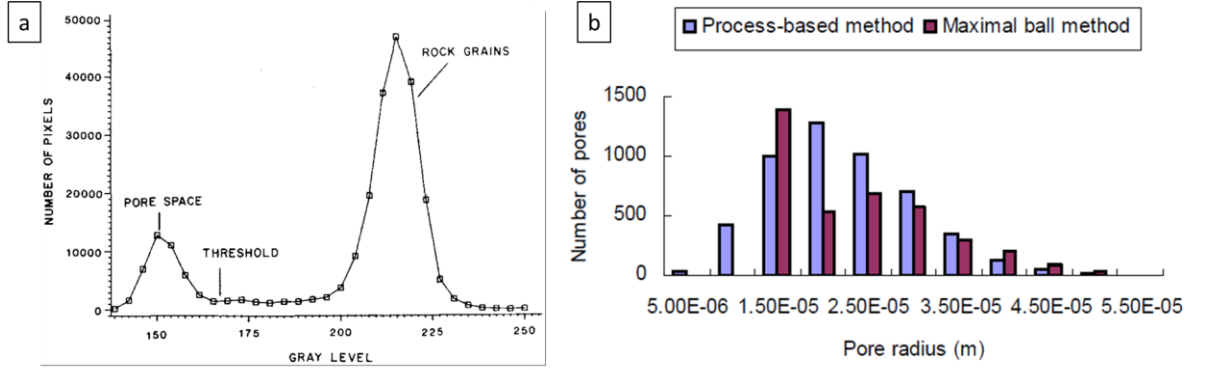


Figure 5: Example of pore size distributions by CT scan. a) legacy pore size distribution (Ruzyla, 1986). b) Recent pore size distribution (Dong et al, 2007).

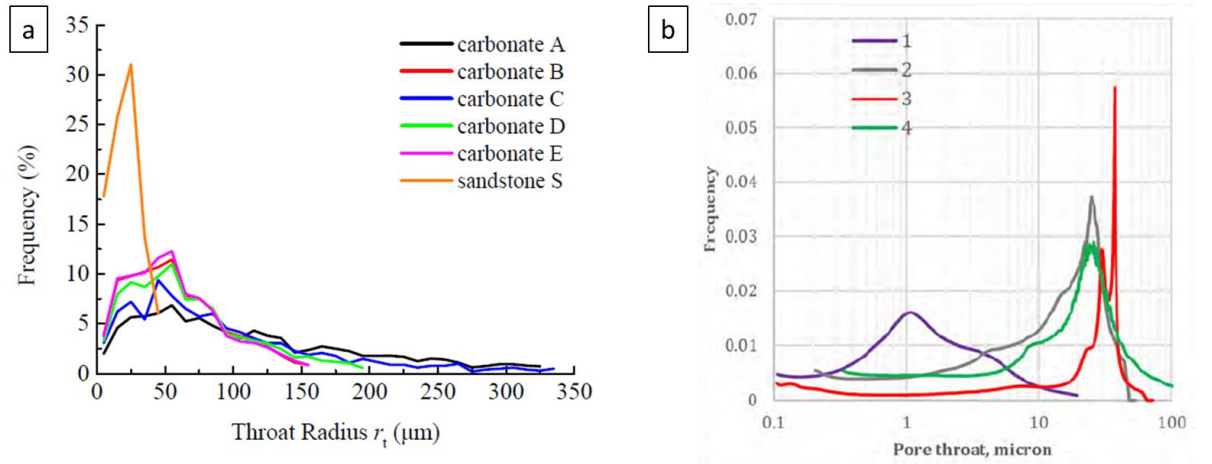


Figure 6: Example of pore size distributions by CT scan. a) (Liu et al, 2022). b) (Gao et al, 2021).

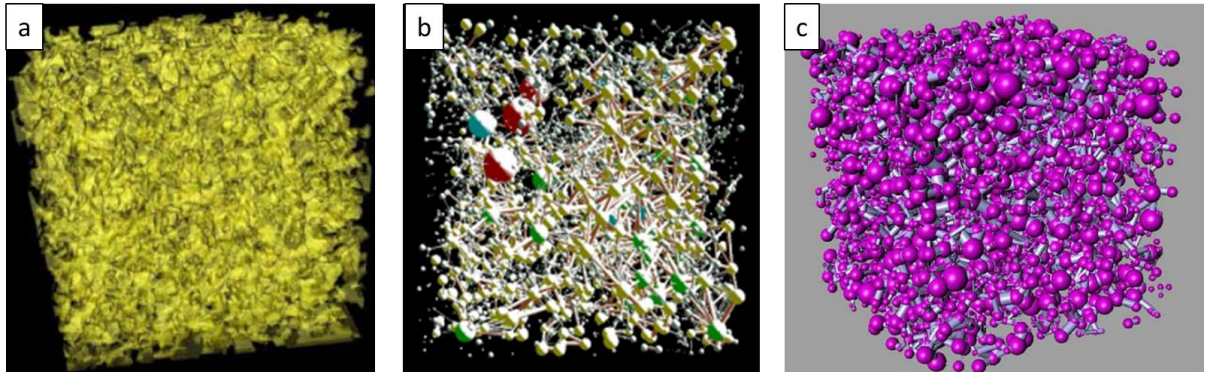


Figure 7: Examples of 3D reconstructed pore models of core samples. a) (Sarker and Sidiqi, 2022). b) (Liu et al, 2022). c) (Dong et al 2007).

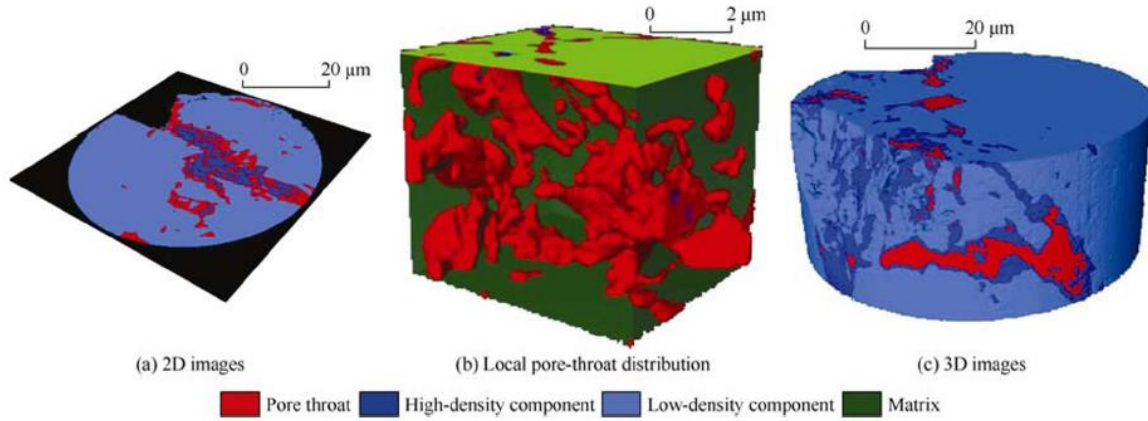


Figure 8: 3D reconstructed model of pore throat at the nano scale using advanced micro-CT scanner. (Bai et al, 2013).

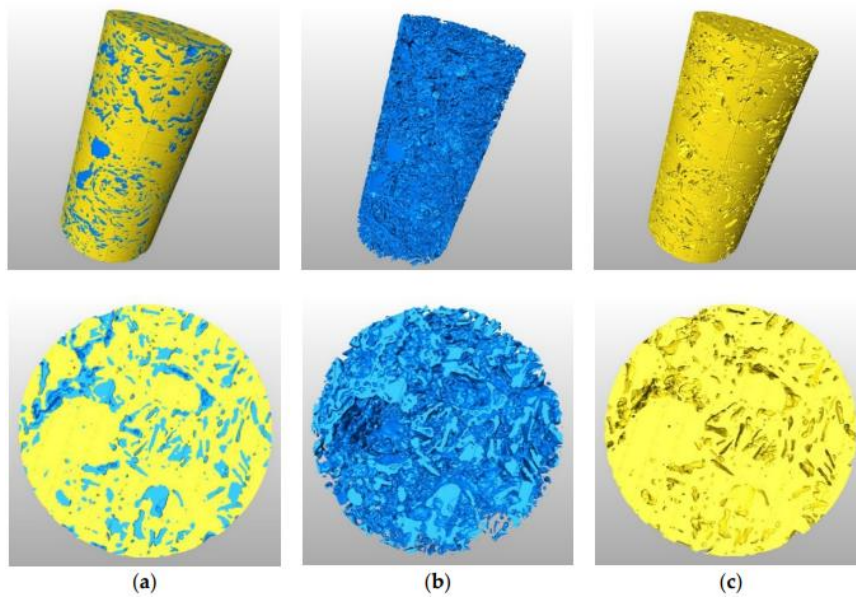


Figure 9: Example of Pore size and throat 3D modeling (Liu et al, 2022) showing models of a) pore size and throat. b) pore throat. c) pore size.

Applications of CT scanning are extended to the characterization of rock samples under dynamic conditions. For example, CT scanning techniques are used to characterize effectiveness of flow in the pore medium. For instance, Zhenpend et al, have studied the

effect of multilayered heterogeneity on the flow by constructing a special CT scan model called “Quasi Static Flow”, then simulating the flow as shown in Figure 10. This technique is used to investigate potential fingering and crossflow scenarios. The drawback of this techniques is that the evaluation of those phenomena is done at extremely small scale, where the degree of representation of the used core sample to the bigger area of investigation is questionable. (2013). Saxena et al study were focused on comparing the simulation results of different microstructure models using CT-Scan, which proves the powerfulness of CT scanning techniques in investigating in-situ flow at localized area (2017). Another useful application of CT scan is the estimation of permeability, where multiple numerical models and correlations were developed to predict the permeability using CT scan measurements. Different permeability models and techniques by CT scan can be found in (Lie et al, 2013), (Saxena et al, 2019) and (Song et al, 2019). Different studies have reported the use of CT scanning techniques to investigate formation damage and mud cake effectiveness such as Elkatatny et al, 2012), (Omar & Nasr-El-Din, 2021), (Van Overvelt et al, 2012) and (Frequin et al, 2012).

Although that CT scan have been proven to be effective to many applications in core evaluation, it comes with limitations. According to Elkhoury (2019), micro-CT scan requires complex and advanced algorithms to obtain a digital reconstruction of the scanned object. In some cases, inversion of micro-CT scans can be limited to the capabilities of the software used, in addition to the capabilities of the micro-CT scanning instrument itself. Another consideration to point is that all CT scanning techniques are done on small areas that may or may not be representation to the actual are of the investigation. For instance, the example abovementioned of the investigation of fingering and crossflow are done in a

2 centimeter of radius core plug to study an area with radius in the magnitude of hundreds meters. This requires intensive evaluation of the representation of the samples used and may need to repeat the experiment on tens of samples to finally be able to draw a good conclusion. Digital storage and size of micro-CT scans and reconstructed models are another challenge that needs to be addressed when dealing with this technology.

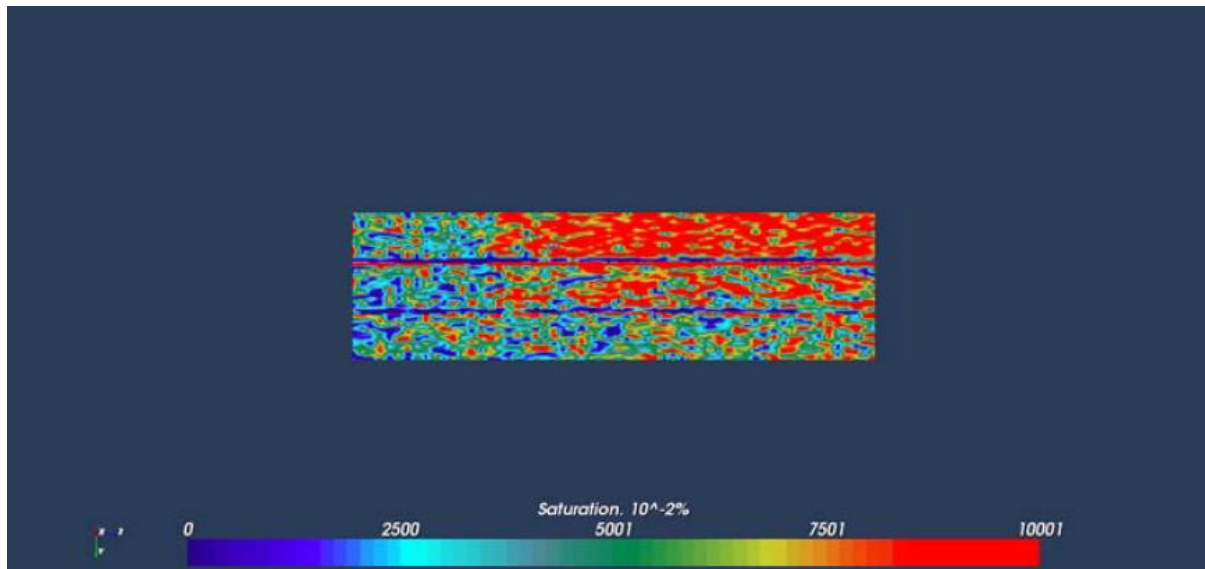


Figure 10: CT Scan image, simulation of flow using "Quasi Stati Flow". (Zhenpeng et al, 2013).

2.2 Nuclear Magnetic Resonance

This section provides a literature review of the use of Nuclear Magnetic Resonance (NMR). The section provides a general overview of NMR and some of the applications and limitations of NMR in the context of the oil and gas industry.

2.2.1 Overview of NMR

Nuclear magnetic resonance has been emerging as one of the most valuable techniques used for formation and fluid characterization in the oil and gas industry. With both laboratory and field applications, the applications of NMR technique have been witnessing

a significant growth. Due to the substantial evolvement of NMR techniques in the past 20 years, NMR became a mainstay logging tool in petrophysical evaluation domains. Historically, NMR is considered to be a product of the evolution chain of the Magnetic resonance imaging (MRI) used the medicine industry. (Dunn et al, 2002).

The utilization of the technique has first arrived in the oil and gas industry as early as the 1960's. Because of numerous limitations, NMR techniques didn't draw much attention at that time and saw almost no success. NMR technique in the oil and gas industry had low profile for 30 years, till 1990's, where the introduction of pulse neutron NMR tool attracted recognition to the capabilities of NMR in the formation evaluation field. Since then, the NMR aquation kept gaining popularity in the industry with continuous enhancements and introduction of multidisciplinary applications. One of the major contributors to the fast risen of NMR is that it is when of the few logs that provides insight into the fluid presence in the porous medium, in addition to dielectric, resistivity, and with less significance density. (Dunn et al, 2002). Beside the quantification of bound and free fluids in the porous media, early pulsed neutron offered more applications such as estimating permeability and porosity independently from the formation density or facies, and the detection and quantification of gas. (Freedman & Heaton, 2004). NMR techniques are built on the concept of exciting the nuclei of atoms by an external magnetic field, causing the nuclei to exhibit a phenomena of spinning.

2.2.2 Applications of NMR

NMR have introduced unique, yet revolutionary capabilities that enabled an improved formation evaluation. One of many and is considered one of the most important NMR applications, is the ability to differentiate between free and bound fluids. Bound fluids (i.e. bound water or clay water) can be defined as the amount of water that would require immense pressure to be produced, and can be identified from the relative permeability curve as the point of which K_b goes to zero. The bound water saturation can be determined from NMR T2 measurement. After the T2 measurement is acquired, a cutoff value is defined depending on the lithology and clay type, signals below the cutoff are considered to be bound fluids. (Singer et al, 2017; Freedman & Heaton, 2004). Figure 11 shows an example of bound fluid estimation. Due to the sophisticated pore body and network shale formation poses, T2 measurements are considered challenging and would require more attention. Testamanity & Rezaee presented a methodology to measure the cutoff values of shale formations, and ultimately estimate the bound fluid saturations. (2019).

Another imperative application of NMR is the pore size distribution. The pore size distribution can be estimated from the T2 NMR measurements of porous system saturated with single phase. Starting with the definition of T2, where it is a function of the relaxation time of surface, bulk and diffusion as given by equation 1. Because in single phase T2 measurement, the surface relaxation time is much faster than the bulk and diffusion relaxations, it is assumed that the T2 measurement is directly proportional to the surface relaxation decay, which can be linked to the size of the pores as given by equation 2, where the relaxation of bulk and diffusion is neglected. (Kenyon et al, 1989; Prammar, 1994; Lu et al, 2021). Figure 12 illustrate an example of multi-porous system pore size distribution.

This technique also can estimate the total porosity of the area of investigation. This can be achieved by calculating the cumulative porosity of the system, which constitute the total porosity.

$$\frac{1}{T_2} = \frac{1}{T_{2\ surface}} + \frac{1}{T_{2\ bulk}} + \frac{1}{T_{2\ diffusion}} \quad (1)$$

$$\frac{1}{T_2} \approx \frac{1}{T_{2\ surface}} \approx \frac{\rho}{r} \quad (2)$$

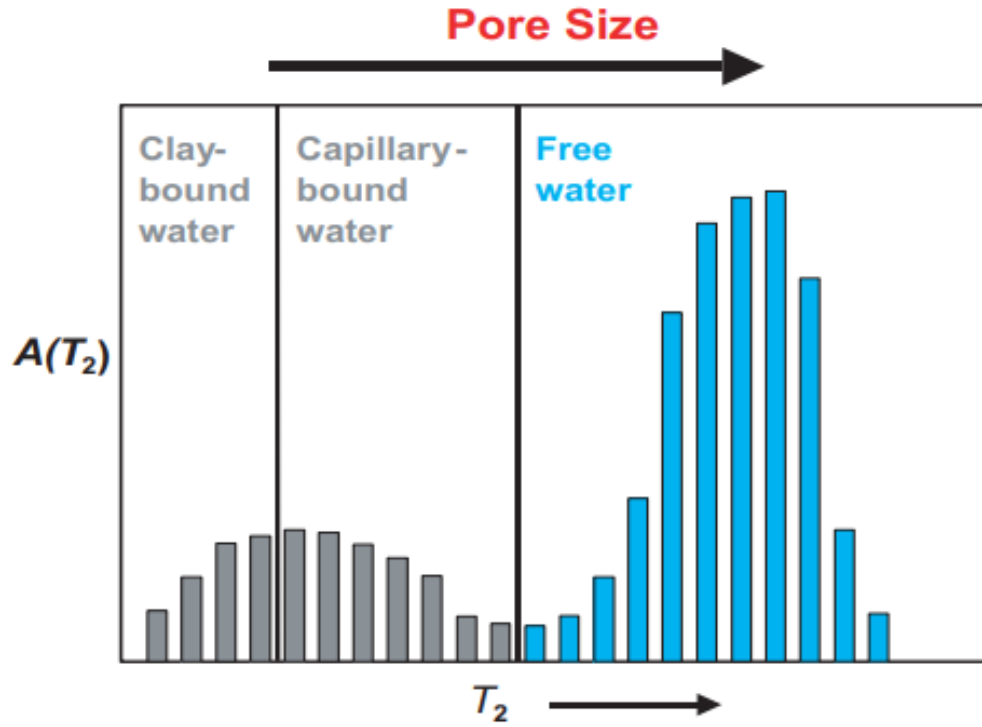


Figure 11: Example of bound fluid estimation from NMR T2 measurement. (Freedman, 2006).

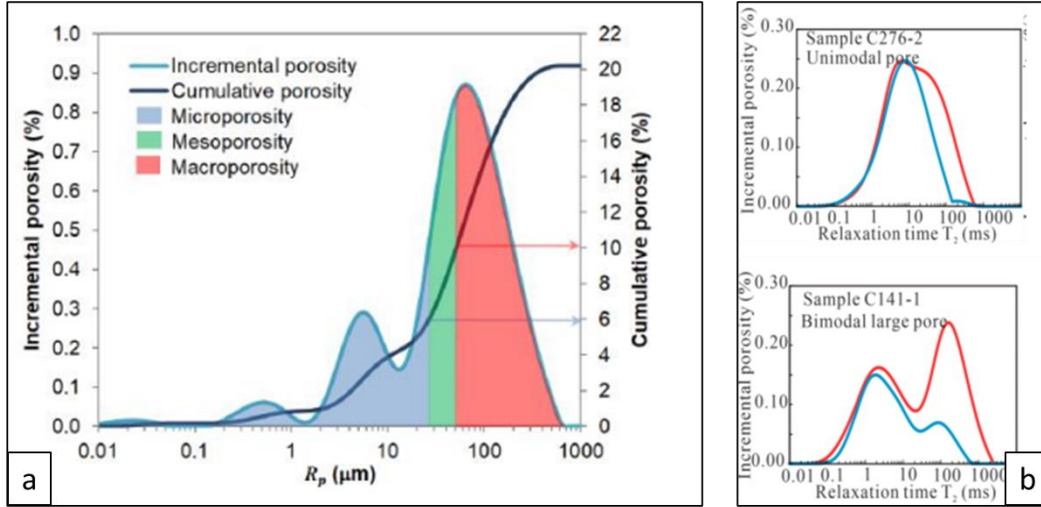


Figure 12: Example of pore size distribution acquired by NMR T2 measurement. A) (Hoerlle, 2018) b) (Lu et al, 2021).

Another useful application of NMR that has been widely used in the industry is the estimation of permeability. Many empirical correlations to estimate permeability from NMR have gained a wide acceptance, as they provide relatively accurate results in quick and cost-effective manner (compared to expensive core analysis). Empirical permeability models for different formations and cases can be found in (Seever, 1966; Kenyon, 1997; Kenyon et al, 1988; Coates et al, 1999, Dunn et al, 2002; Mao et al, 2013; Wei et al., 2016). Due to the wide acceptance of NMR in predicting permeability, researchers started to look at it in more complex and tight formations such as shales and source rocks. Recent study showed the effectiveness of NMR in estimating permeability in tight formations when compared to pulse-decay technique, as shown in Figure 13. (Shen et al., 2021). At core scale, NMR measurement can provide valuable insight about the rock sample quality and can be a useful tool of characterization. As NMR measurement at core sample scale is often used to characterize the modality of the porous medium. NMR also provides a valuable assessment of permeability and the degree of the sample's heterogeneity and anisotropy.

Due to the ease and quickness of NMR core measurement, NMR is often used along with conventional core analysis (routine core analysis) to screen samples and optimize the selection of core samples for more complex and time consuming SCAL experiments such as relative permeability and Amott Wettability. (Evan R. McCarney et al., 2015; Moss, 2004). Some publication reported the use of NMR in evaluating the effectiveness of filter cake formation and removal, as illustrated in Figure 14. (Adebayo & Bageri, 2020; Bageri et al., 2021).

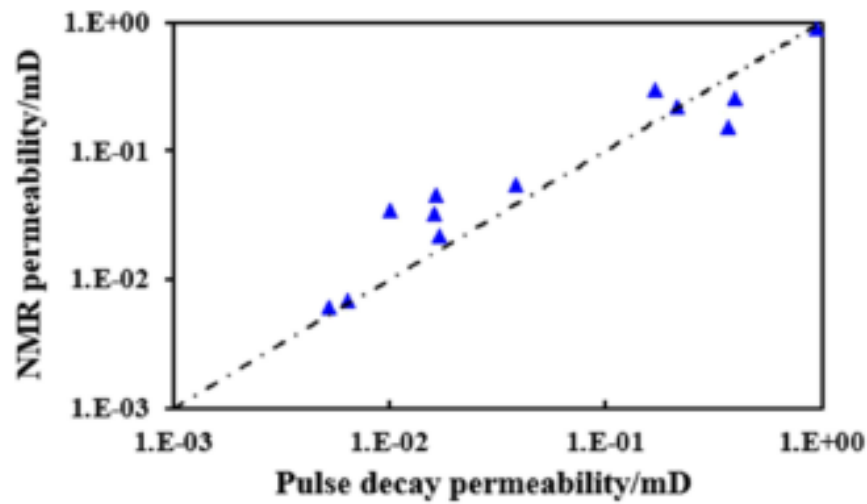


Figure 13: Comparison between NMR and Pulse-decay in estimating permeability of tight formation. (Shen et al., 2021)

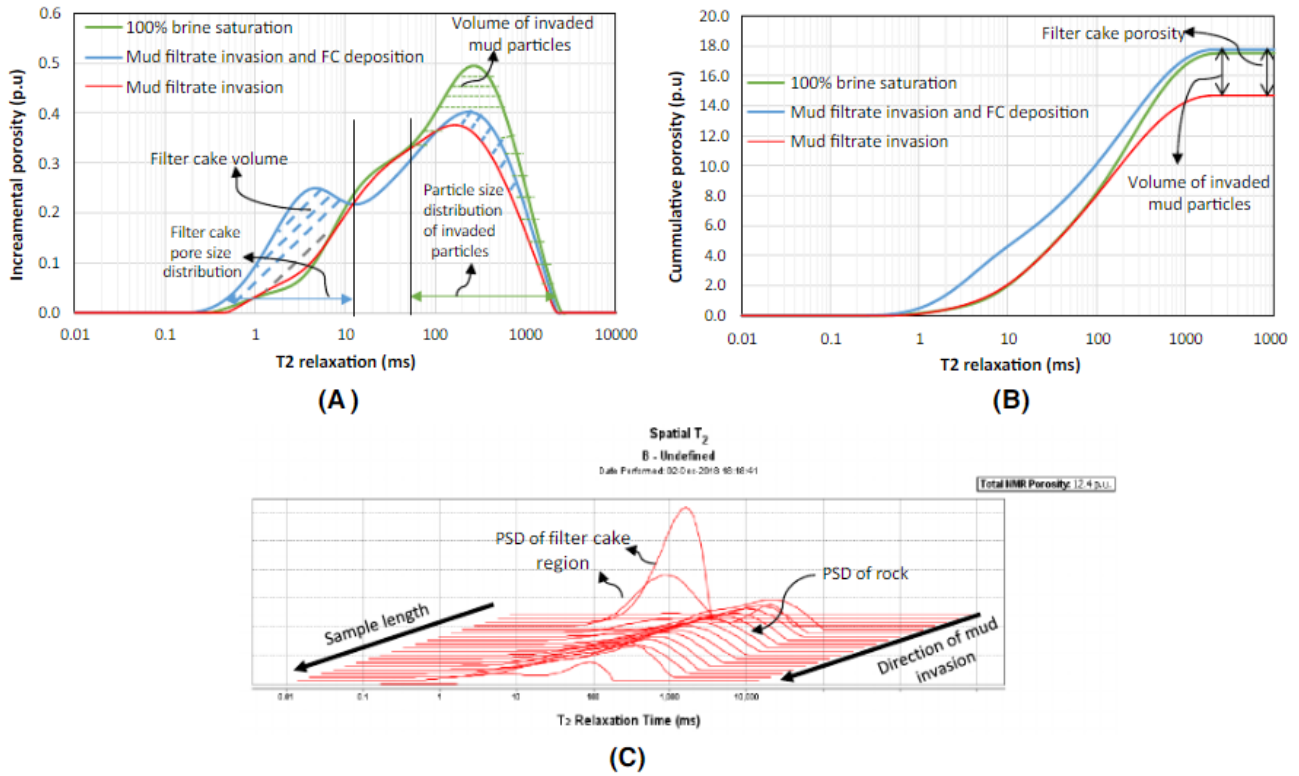


Figure 14: Illustration of how NMR is used to evaluate the effectiveness of mud cake removal. (Adebayo & Bageri, 2020)

Wettability measurement, among other essential parameters had an accelerating development in the NMR field. Different techniques have been developed and established to quantify wettability. However, wettability by NMR is still a field of development in the research. The focus of research in NMR is recently moving toward field measurement of wettability. The essence of NMR is built on the relaxation processes, spin-lattice relaxation (T_1) and spin-spin relaxation (T_2). The T_2 relaxation is a combination of bulk relaxation (T_b), surface relaxation (T_s) and diffusion relaxation (T_D).

The first NMR technique to quantify wettability is T_2 Wettability technique as given in equation 3 and equation 4 where S_r denotes the effective surface. The drawback of this technique is that it is dependent on several other sources of information (e.g. saturation,

porosity, density). However, this technique gave a comparable results to USBM as shown in Figure 15. (Valori, 2018; Looyestijn, 2006, 2007; Al-Muthana, 2012; Freedman, 2012). Similar to this approach but in a simplified fashion, Al-Mahrooqi technique (T2 saturation-based), which assumes that the saturation is with higher significance than other parameters. This technique assumes that the effective surface (S_r) is zero, therefore the spin-spin relaxation will be a function of bulk relaxation only as in equation 5. (Al-Mahrooqi, 2003).

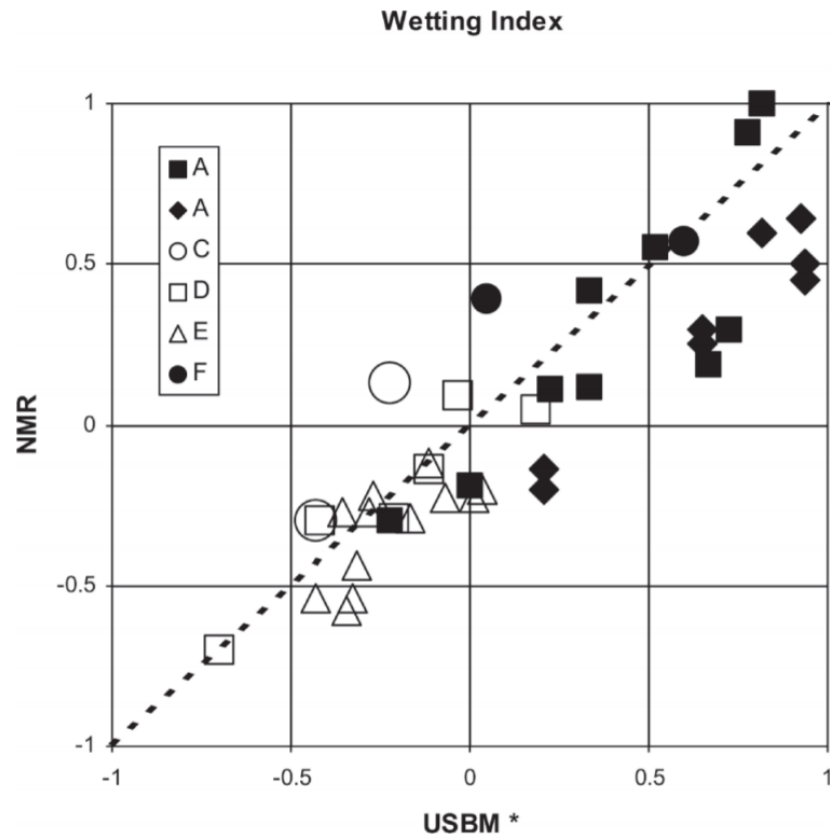


Figure 15: Comparison between the NMR T2 wettability technique and USBM. (Looyestijn, 2006).

$$\frac{1}{T_{2,brine}} = \frac{1}{T_{b,brine}} + \rho_{brine} \frac{S_{r,brine}}{V_p S_{brine}} \quad (3)$$

$$\frac{1}{T_{2,oil}} = \frac{1}{T_{b,oil}} + \rho_{oil} \frac{S_{r,oil}}{V_p S_{oil}} \quad (4)$$

$$\frac{1}{T_2} = \frac{1}{T_b} \quad (5)$$

Another NMR technique for wettability measurement is diffusion. Hurlimann proposed a model (1994) to measure wettability from NMR. His model was a function of too many parameters (e.g. tortuosity, pore size, apparent relativity, etc.), therefore it was complex and it is associated with an inherent error. Recently, a workflow to determine the wettability was developed based on the diffusion-T2 map. This is done by locating water and oil in the diffusion-T2 map as illustrated in Figure 16. This technique estimates the wettability primarily based on the inversion of diffusion data to surface relaxivity or both water and oil. Similar to previously discussed NMR methods, this technique requires the acquisition of additional information apart from diffusion and NMR-related data. Moreover, this technique is not functional for field evaluation with wells drilled with oil-based mud. (Minh, 2015).

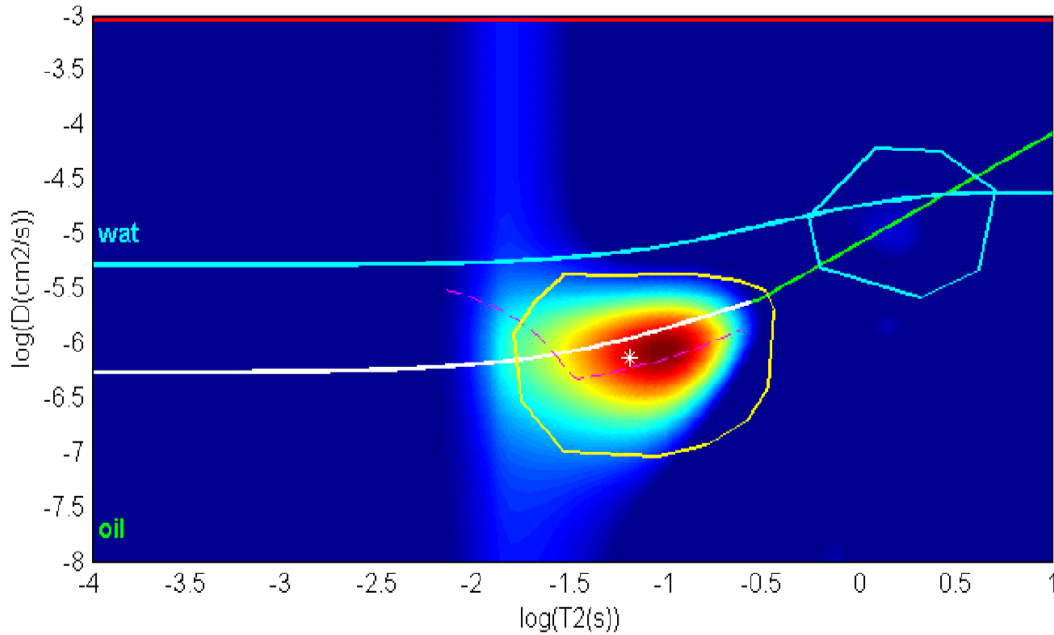


Figure 16: Example of water and oil on a diffusion T2 map. (Minh, 2015).

There are many other NMR techniques and approaches to determine the wettability, most of which are associated with many challenges and difficulties. One of those is the T1/T1 ratio technique, which provides a wettability measuring model with high correlation to standard tests such as USBM. However, implementation of this model consumes a huge amount of time, which is not tolerable by any commercial lab let alone by field operations. (Valori, 2017).

2.2.3 NMR Diffusion Coupling

For pore size typing and as explained in section 2.2.2; the estimation of pore size distribution relies on the sole proportionality between T_2 and surface relaxations (equation 6). Extending this assumption to a dual porosity model implies that the fluid particles have simultaneous relaxation and diffusion between the two pore bodies. Moreover, the complexity increases when multiple fluids coexist at the pore microscopic level, which is the case for most NMR field logging with the presence of drilling fluid filtrate, reservoir hydrocarbons and formation brine. In such a case, NMR T_2 response is dependent on surface relaxivity, pore structure, fluid's free bulk relaxation as well as fluid diffusivity. Thus, modeling NMR T_2 solely based on surface relaxivity might be misleading. Diffusion coupling phenomena is a proof to that, where it occurs in interconnected dual continuum porous mediums, where fluid molecules diffuse interchangeably through the connected porous bodies. In ideal cases (mostly in dual isolated porosity), fluid particles diffuse independently in each isolated porosity body causing T_2 signal to capture the two porosity models. However, in non-isolated porosity bodies, because the fluids phase has a continuous connection across both bodies, the fluid's diffusion is exchangeable which

results in an average relaxation representing a pseudo unimodal in between the two models as illustrated in Figure 17. If used, the pseudo average unimodal might result in deceptive petrophysical properties such as irreducible water saturation and permeability. Several studies have investigated diffusion coupling under the presence of single-phase fluid.

Brownstein and Tarr have characterized the NMR pulse decay in coupled porous medium by the introduction of three variables: Brownstein number (μ) which is defined in equation 6, pore aspect ratio (η) and microporosity fraction (β). (Brownstein and Tarr, 1979).

$$\mu = \frac{\text{relaxation rate}}{\text{diffusion rate}} = \frac{\rho L}{D} \quad (6)$$

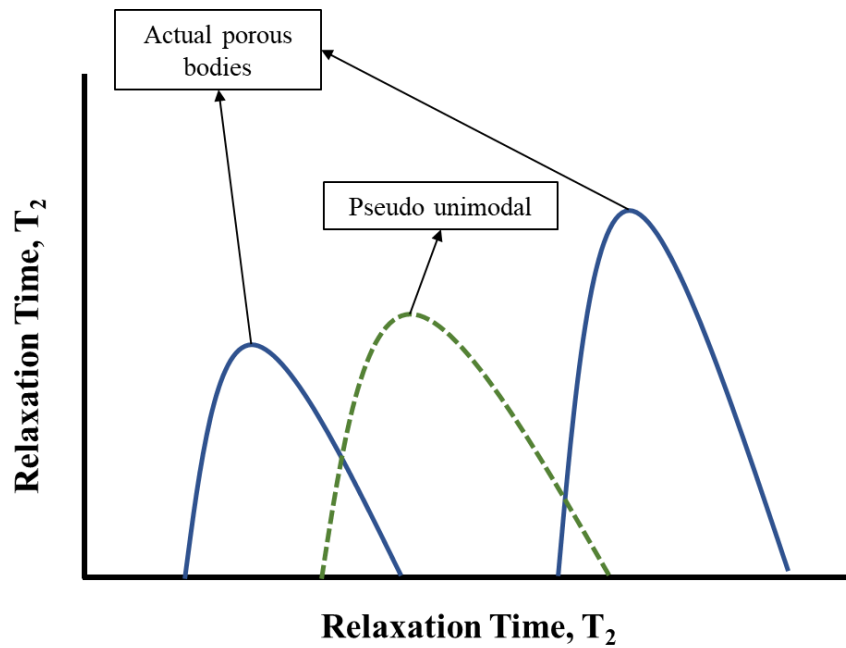


Figure 17: Example of diffusion coupling phenomena

As μ was introduced for a single dimensional analysis and assumed a fixed surface to volume ratio, it failed to model the relaxation of bimodal porosity system. Anand and Hirasaki presented a more realistic coupling characterization by the introduction of coupling parameter (α) given by equation 7. Their numerical simulation study concluded

that as α increases, the model exhibits a stronger dual porosity characteristics as shown in Figure 18. Their numerical simulation relies on inputs obtained from T_1 and T_2 relaxation at 100% water saturation and produces an averaged value of α . Figure 19 provides a comparison between the simulated and experimental results of pore size distribution. (Anand & Hirasaki, 2007).

$$\alpha = \frac{1/T_{2c}}{D/L_2^2} \frac{\rho \beta L_2^2}{D L_1} \quad (7)$$

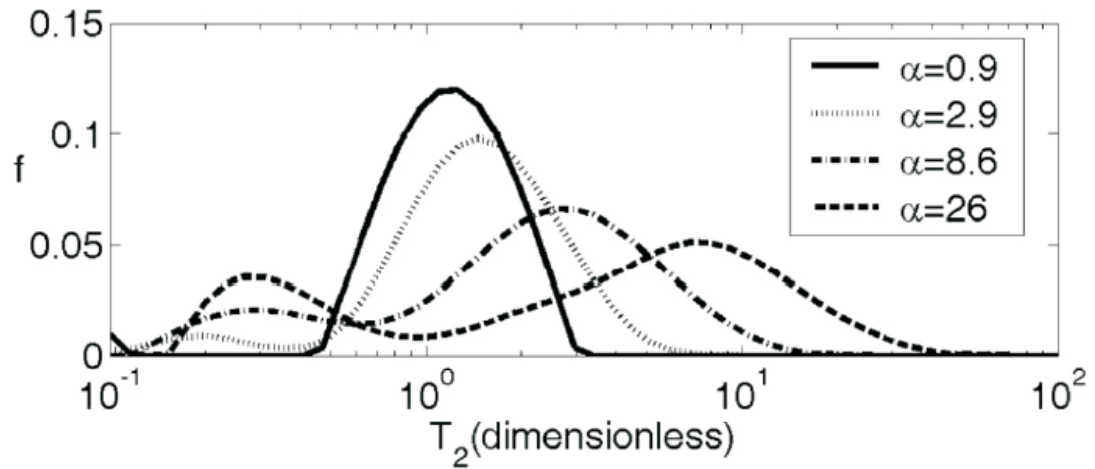


Figure 18: Simulated T_2 distribution illustrating the effect of the presence of dual porosity model on α . (Anand & Hirasaki, 2007).

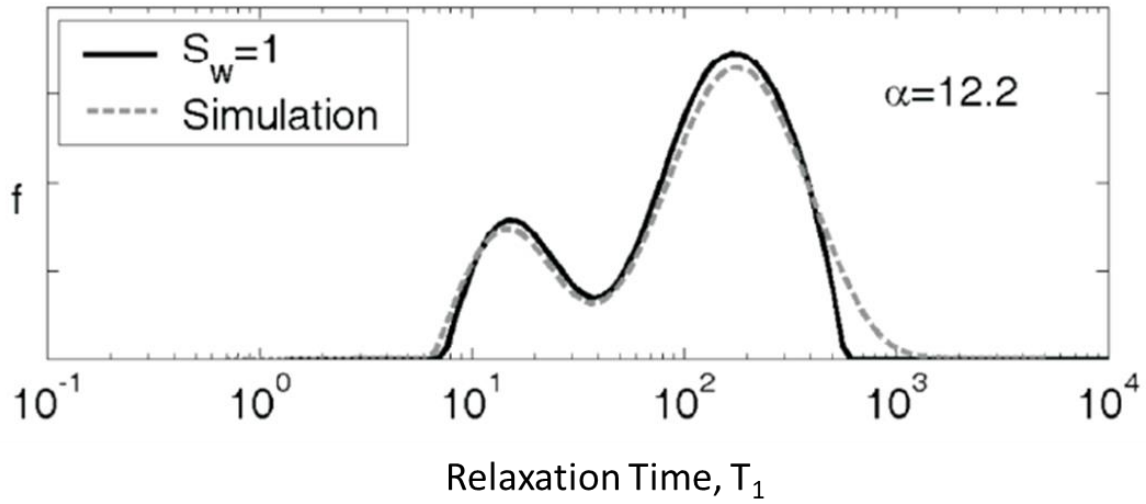


Figure 19: Comparison between simulated and measured T_1 showing a good match. (Anand & Hirasaki, 2007).

Fleury & Soualem in 2009 have studied the diffusion coupling by a 2D T_2 technique with a storage time. They used shale sample to study the diffusion in multimodal system with extremely tight porosity. They introduced the T_2 - T_2 storage technique to evaluate the coupling. In T_2 - T_2 measurements of two distinctive pore systems, at an early storage time, signals present the initial position of protons and are aligned with the diagonal line, indicating the distribution of the pore structure. At a later storage time, off-diagonal peaks might emerge with the movement of the initial signal peaks across the diagonal line, indicating the strength and direction of the exchange between the two pore systems, as illustrated by XX. Additionally, building on the work done Anand & Hirasaki, they have introduced a dimensionless factor to characterize the diffusion coupling and used simulation to evaluate the results, where XX provides an example of the simulation work they achieved with a good accuracy compared to MICP. The approach involved complex math where values are solved then inverted from the Laplace domain.

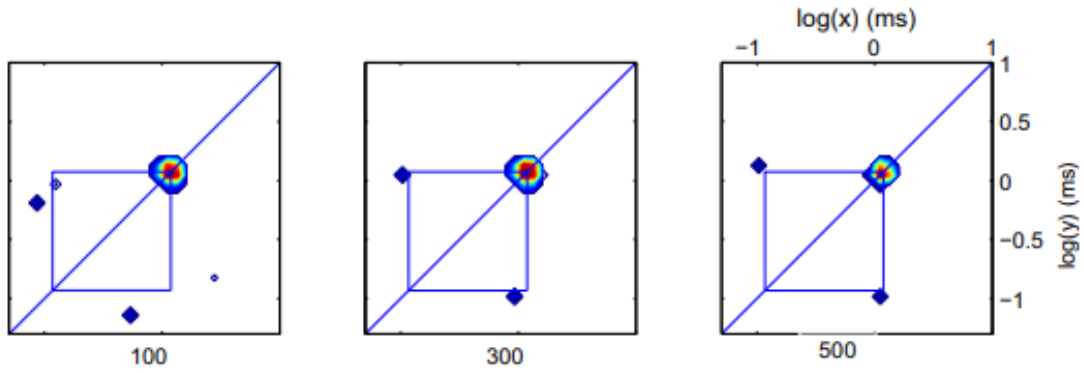


Figure 20: Illustration explains the utilization of 2D T2 storage to evaluate coupling. (Fleury & Soualem, 2009)

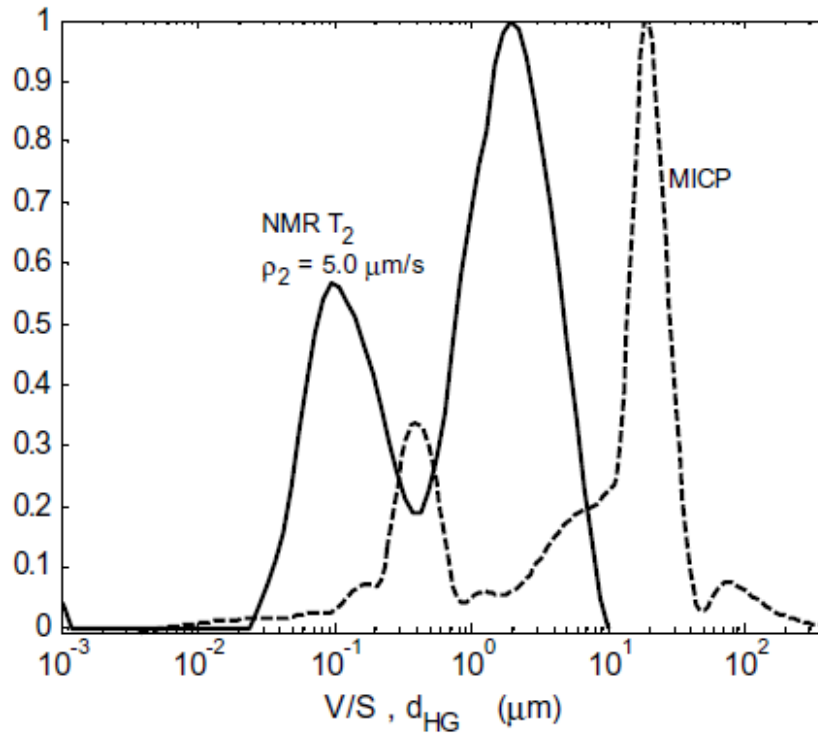


Figure 21: Simulation modeling showing a good comparison to MICP data. (Fleury & Soualem, 2009)

Toumelin et al. (2002), have developed a randomized simulation model that utilizes Monte-Carlo random walks to evaluate the diffusive nature of reservoir fluids (oil and

water), and their effects on the strength of coupling. Their study revealed the diffusive nature of the dual porosity system they studied, under a single saturation of oil and water. They reported that diffusion coupling significance on the T_2 signal increases when: (1) the system is characterized by the presence of microporosity. (2) the connectivity between macro and micro pore body is well established. (3) Rock surface relaxivity is low, enabling diffusion to become more dominant. (4) Temperature solicits and excites protons, resulting in increased diffusion. Figure 22 presents the findings of temperature effect on diffusion.

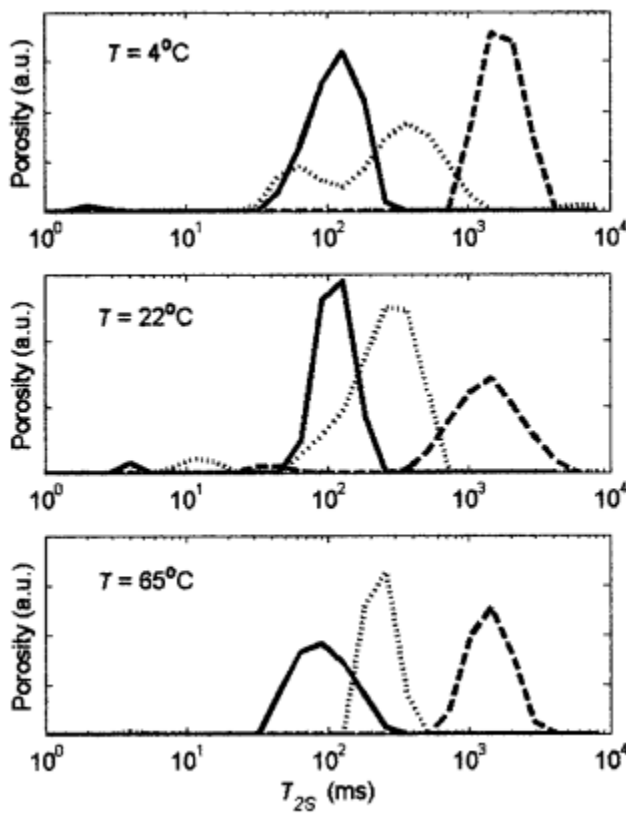


Figure 22: Simulation study reported that temperature increase excites the protons resulting in higher diffusion. (Toumelin et al. et al., 2002).

Grunewald and Knight have examined the diffusion coupling on pure synthetic gel silica samples with a strong dual porosity characterized as intragranular microporosity and intergranular microporosity. The focus of their study was to investigate the significance of geochemistry on diffusion coupling. They examined the diffusion coupling effect on identical samples with different concentrations of adsorbed paramagnetic (Fe^{3+}). They observed that controlling the geochemistry of surface relaxation by increasing the concentration of absorbed paramagnetic that coats the surface, produces improved and more distinctive amplitude peaks representing two pore bodies, yet not capture the true distributions, as shown in Figure 23. (Grunewald & Knight, 2009). Table 1 provides a high level summary of key papers that studied diffusion coupling.

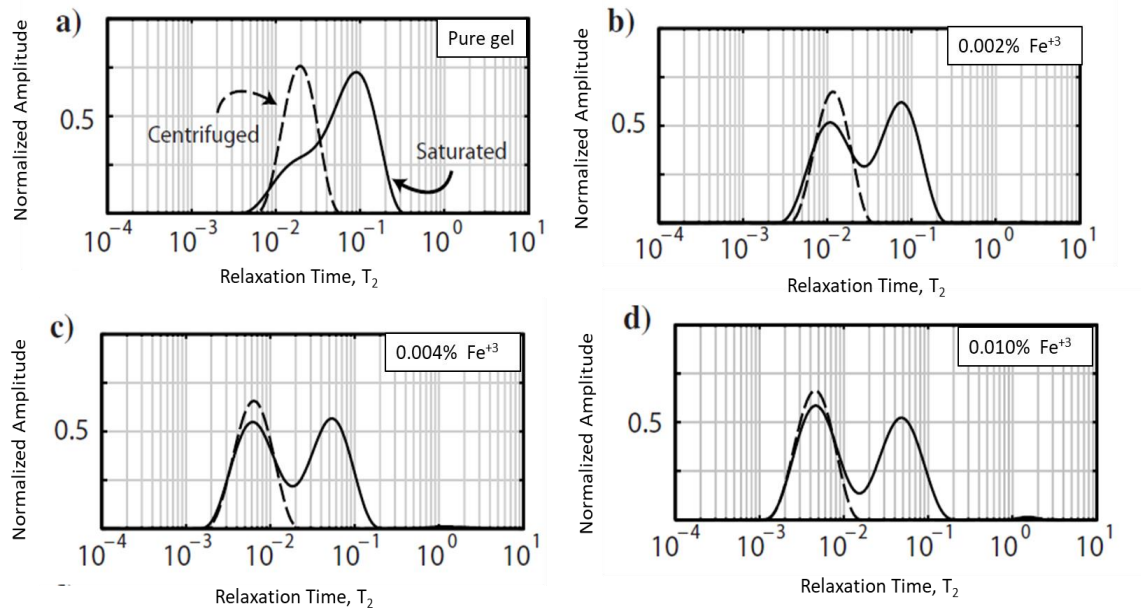


Figure 23: Effect of paramagnetic concentration on diffusion coupling. a) pure silica sample. b) sample with 0.002% Fe^{3+} . c) sample with 0.004% Fe^{3+} . d) sample with 0.010% Fe^{3+} . (Grunewald & Knight, 2009).

Table 1: Summary of key papers that evaluated diffusion coupling

Paper	Examined factor	Summary	Limitations and shortcoming
(Brownstein and Tarr, 1979).	Pore geometry and diffusivity	Introducing the concept of diffusion coupling, characterized by Brownstein factor	Conducted the analysis on synthesized sample and single-phase water only. Did not consider the other effects
(Anand & Hirasaki, 2007).	Pore geometry and diffusivity	Used simulation to evaluate coupling and introduced the coupling parameter (α)	Simulated the analysis for single-phase water, with no experiments to support the work.
(Grunewald & Knight, 2009):	Pore geometry and surface relaxivity	Studied diffusion coupling in dual porosity Silica Gel system and used Ferromagnetic cations to decouple the system	Did not consider diffusion coefficient, or bulk diffusion. Their work was limited to single-phase. Their solution has no field applications.
(Fleury & Soualem, 2009)	Pore geometry and diffusivity	Studied and confirmed the diffusion coupling in dual-porosity shale samples, used 2D T2 for evaluating the coupling	Their work involved complex math, and was not validated with experimental.

CHAPTER 3

METHODOLOGY

The methodology of this study is centered around experimental laboratory analysis to investigate the diffusion coupling phenomena under the presence of multiple fluids, complemented by NMR simulation. This section details the methods, material and approach followed to attain the presented results.

3.1 Approach

This work presents a focus on fluid's diffusional coefficient on diffusion coupling. To establish a comprehensive understanding, during the course of this work other factors that are tangible to diffusion coupling, such as pore structure and grain size were also evaluated. For a Sift analysis and understanding of the governing factors related to the phenomena, Silica Gel samples were used as a control medium. As such samples allows for to study the effect of varying important parameters such as pore size and grain size, in a controlled settings. More complex, yet more representative samples, which are core plug samples obtained from Saudi Aramco, were also used to vet and check the conclusions drawn from the control media. Figure 24 presents the workflow followed for each sample to achieve the work.

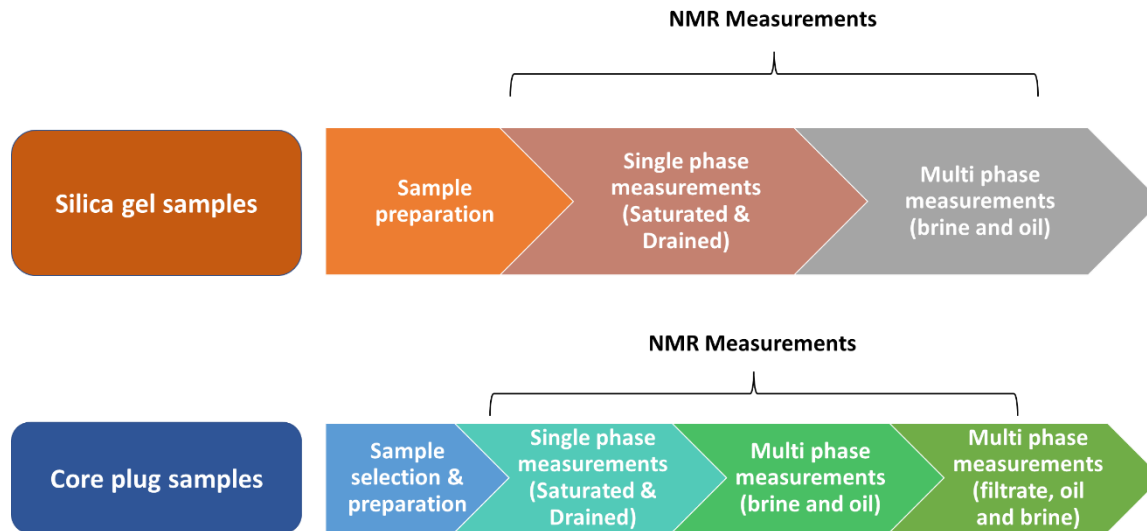


Figure 24: Experimental workflow followed for Silica Gel Samples & Core plug samples to achieve the results

3.2 Materials and Preparation and Measurements

The materials and methods used in this study are presented in this section. It provides an overview of fluids, materials, and core plug samples along with the equipment used in the experimental work. Additionally, the procedure and methodology followed to obtain the results and achieve the objectives of this work are detailed.

3.2.1 Fluids

Multiple fluids have been used to investigate the extent of the effect of the fluid's diffusion coefficient on the diffusional coupling. Deionized water, Saline water and Hydrofluoric acids with different weight concentrations were obtained. Fluid properties, including density, dynamic and kinematic viscosities were measured using Anton Paar SVM 3001 Stabinger viscometer, as shown in Figure 25. Three measurements were taken, and the average values are reported to ensure repeatability and accuracy, where all measurements were in acceptable range. Table 2 shows the properties of water and Hydrofluoric acids.

Table 2: Summary of water and HCl fluids' properties

Label	Density (g/cc)	Dynamic Viscosity (mPa.s)	Kinematic Viscosity (mm²/s)
5% HCl	1.021	0.969	0.949
10% HCl	1.047	1.052	1.005
15% HCl	1.072	1.136	1.059
Deionized water	0.997	0.999	1.002
Saline Water	1.022	0.873	0.854



Figure 25: Anton Paar SVM 3001 Stabinger viscometer used to measure fluid properties

Additionally, four different crude oil samples resembling extra light, light, medium and heavy oil are obtained. Diesel, which is often used as the base fluid in oil base mud system, was used to investigate the effect of drilling filtrate presence. Properties of fluids used in the study are presented in Table 3.

Table 3: Summary of the properties of crude oil samples and Diesel

Label	Density (g/cc)	Dyn. Visc. (mPa.s)	Kin. Visc. (mm²/s)	γ (API)	Asphaltene content (%)	T_{2,peak} (ms)	T_{2,LM} (ms)
Diesel	0.820	3.59	4.38	41.06	-	562	378
Extra Light	0.819	2.97	3.63	41.27	-	794	426
Arabian light	0.852	7.13	8.38	34.57	6.83	282	112
Medium	0.868	14.40	16.59	31.44	9.19	112	37
Heavy	0.884	28.22	31.91	28.43	13.5	71	22

3.2.2 Silica Gel

Multiple synthetic Silica Gel samples were obtained and used in this work. The purpose of the various samples is to investigate the effect of grain size and pore structure on diffusion coupling. Those samples consist of two sizes of grain, to create two system porosity, micro and macro. Table 4 presents the properties of the Silica Gel samples. Figure 26 shows an example of Silica Gel Samples used. Figure 26 shows an imaging of a Silica Gel sample obtained by a Scanning Electron Microscope (i.e. SEM).

Table 4: Summary of the properties and labels of Silica Gel Samples

Sample label	Larger grain size (μm)	Smaller grain size (μm)	Pore size ($\text{\AA}$)	Pore size (μm)
G (100 – 300) P (500 $\text{\AA}$)	100	300	500	0.05
G (315 – 500) P (150 $\text{\AA}$)	315	500	150	0.015
G (70 – 200) P (150 $\text{\AA}$)	70	200		
G (35 – 70) P (150 $\text{\AA}$)	35	70		
G (1000 – 3000) P (60 $\text{\AA}$)	1000	3000	60	0.006
G (70 – 200) P (60 $\text{\AA}$)	70	200		
G (90 – 130) P (60 $\text{\AA}$)	90	130		
G (35 – 70) P (60 $\text{\AA}$)	35	70		
G (20 - 45) P (60 $\text{\AA}$)	20	45		



Figure 26: Example of the Silica Gel samples used in this work, manufactured by ROTH company.

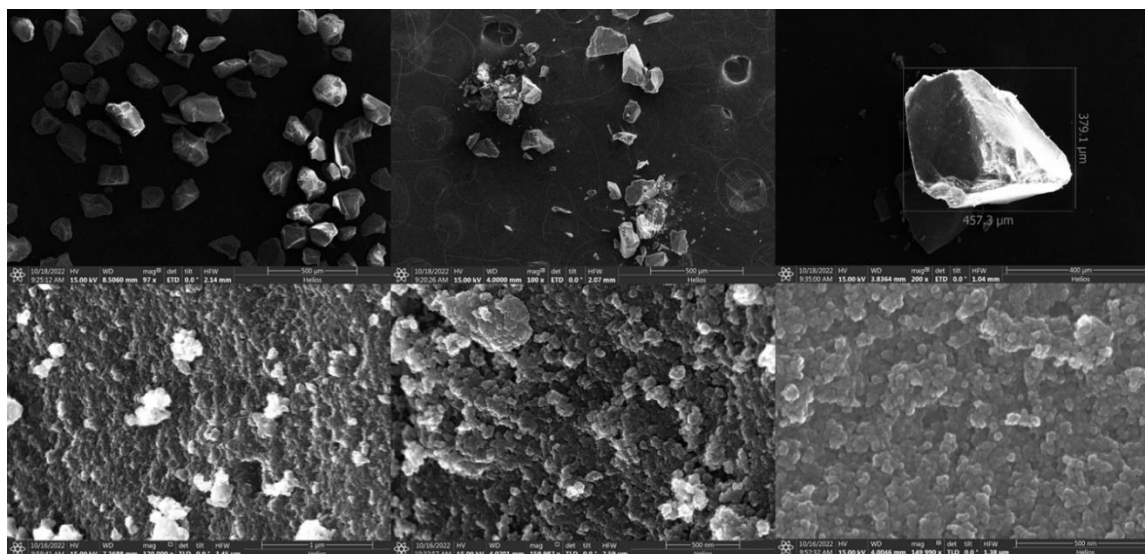


Figure 27: SEM imaging of the silica gel sample.

The following procedure was followed to saturate each sample, in preparation for the single-phase NMR measurement. Figure 28 shows a schematic of the setup while Figure 29 shows an example of samples placed in the desiccator under vacuum.

1. Take the NMR background measurement of the empty glass bottle (that will be used in the following steps) with the cover closed. This is to ensure that the sample measurements are not affected by the container signal.
2. Take the measurement of the inner diameter of the bottle to use later for bulk volume estimation, which will be inputted for porosity base calculation.
3. Have fill the bottle with the Silica Gel sample.
4. Place the Silica Gel sample inside a vacuum glass desiccator.
5. After applying grease, close the desiccator cover and connect it to a vacuum pump.
6. Ensure that the system is well vacuumed (pressure gage may be used with a reading of >20 psi).
7. Leave under vacuum for 24 hours to eliminate remaining air in the sample.
8. Pore the desired fluid using the suction pressure generated by the vacuum pump into the glass bottle.

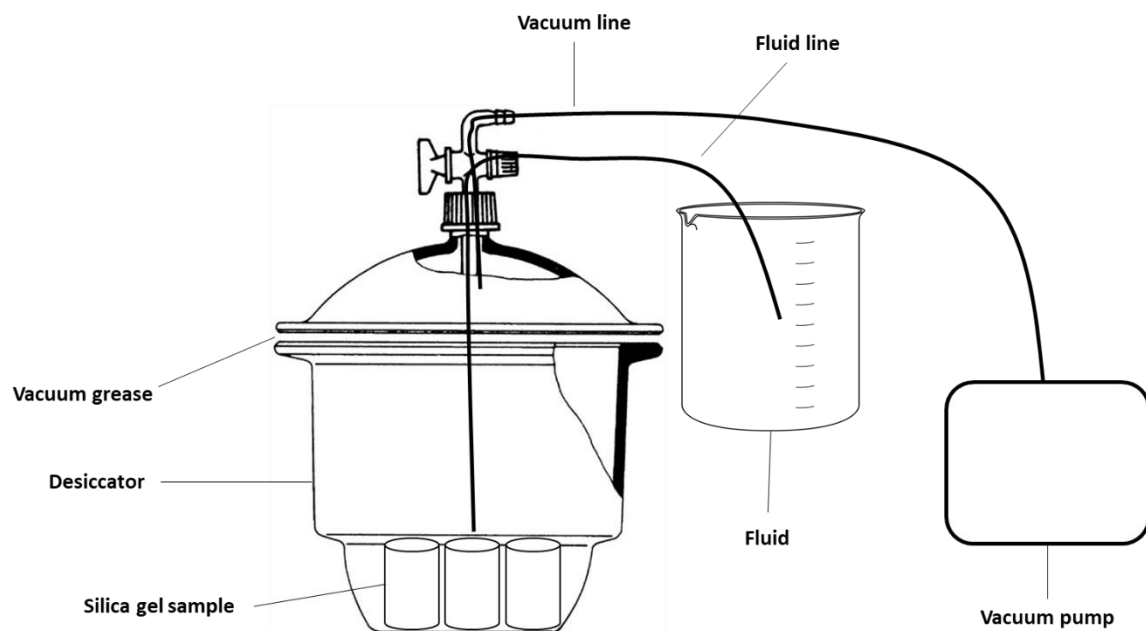


Figure 28: Illustration of saturation desiccator setup



Figure 29: Silica Gel samples prepared for saturation by placing inside vacuum desiccator.

The following procedure was followed to desaturate the samples. It is worth noting that NMR T_2 measurements were taken for each sample after each step to monitor desaturation progress.

1. Complete all of the fully saturated NMR measurements.
2. Open the glass bottle's cover and place the sample under fume hood for 48 hours.
3. Desaturate the samples using a three-micron filtration paper.
4. Observe desaturation progress by the change between the NMR time-lapse measurement and cumulative porosity.
5. In case there is no progress in the desaturation (this was observed with oil samples), utilize a sieve mesh with a size of 36 micron to complete the filtration. Measure NMR daily and observe the changes.

As Silica Gel samples are Silicon Dioxide based material, which is the primary constituent of quartz, they exhibit a water-wet character (Ali et al., 2021). Wettability alteration to oil-wet was attempted to investigate the extent of surface relaxivity, which can be linked to wettability. The following procedure was followed to alter wettability:

1. Saturate the sample with single phase water by following the saturation procedure aforementioned.
2. Desaturation of the sample by following the drainage procedure mentioned earlier to achieve the S_{wi} . This is to ensure that the macro pore body is desaturated, and micro is filled with water.

3. Saturate the sample under vacuum (using the desiccator) with the wettability alteration chemical. The purpose of this step is to alter the wettability character of the macro pore structure of the sample, while the macro pore would still be water wet and filled with water.
4. Age the sample at 60 °C for 120 hours.
5. Desaturate the remaining chemicals by the aforementioned desaturation procedure.
6. Saturate the sample with oil under vacuum. After this step, it is assumed that the sample is filled with water in the micro pore body while filled with oil in the macro, that is now oil wet.
7. Measure NMR T_2 .

3.2.3 Core Rock Plug

Multiple carbonate core rock plug samples of 1.5 inches in diameter were obtained for the study, as shown in Figure 30. This section details the preparations, properties and procedures used with the plug samples to obtain the results presented in the next section. Fundamental properties were measured on the samples as received (clean and unsaturated condition). Rock diameter and length were measured using a digital caliper micrometer. conventional core analysis (CCA) where conducted to determine grain and bulk density, pore-volume, porosity, and permeability using helium porosimeter (AP-608). Boyle's law will be used to calculate the porosity from the acquired data. Whereas Klinkenberg's gas slippage correction will be applied to estimate the permeability. Table 5 presents rock plug samples fundamental properties. Samples were then saturated with water using a desiccator

and aged for +7 days to reach an equilibrium in the grain surface level, in preparation for NMR base-line measurements.

Table 5: Summary of the properties of the core plug samples used in the study

Sample #	Diameter (mm)	Length (mm)	Dry Weight (g)	Dry Weight, after heat shrink (g)	Bulk Volume (ml)	Grain Volume (ml)	Pore Volume (ml)	Porosity (%)	Permeability (mD)
1	38.24	37.29	97.42	102.30	42.83	35.934	6.561	15.32	117.75
2	38.28	30.28	77.34	81.32	34.85	28.38	5.961	17.10	6.84
3	38.18	34.25	93.06	97.55	39.21	34.329	4.551	11.61	57.99
4	38.13	34.52	87.83	92.41	39.42	32.461	6.311	16.01	0.44
5	38.25	38.07	99.28	104.32	43.75	36.651	6.813	15.57	76.59
6	38.21	35.21	92.71	96.98	40.37	34.235	5.741	14.22	0.13
7	38.26	35.70	96.64	101.37	41.04	35.648	5.064	12.34	17.93
8	38.24	30.16	81.57	85.58	34.64	30.054	4.148	11.97	1.15
9	38.03	35.10	89.32	94.00	37.8	32.966	6.318	16.71	0.16
10	38.16	32.73	81.00	85.37	37.43	29.703	6.644	17.75	5.94



Figure 30: Core plug samples obtained for the study.

Mercury injection capillary pressure (i.e. MICP) measurements were performed on sister core plug samples of 1” diameter. MICP can provide an insight into the pore throat and pore body structures and distributions of the samples. (Clerke et al., 2008) and (Al-Awdah et al., 2017). The sister samples may have some variation from the core plug samples used in the study. As a control check, helium porosity were used to compare and evaluate the degree of representation of MICP to the actual core plug samples.

URC-628 Ultra Rock Centrifuge was used at rotational speeds of 1,000 – 12,000 RPM to desaturate the plug sample and establish S_{wi} . Furthermore, it was used to inject oil into the samples.

3.2.4 NMR Measurements

Three different NMR measurements were utilized: NMR T2 measurements (T_2 - T_2), 2D NMR measurements and NMR pulsed field gradient (PFG). One of the most used NMR techniques for both laboratory and field logging applications is the T2 measurement as it provides valuable insights of pore structure, and it enables the estimation of both bound fluid and permeability. In addition to the information it provides, T2 NMR measurement

popularity is owed to fast time acquisition compared to T2 mode. Early work by (Brownstein and Tarr, 1977) revealed that the T2 relaxation measurement can be modelled as:

$$\frac{1}{T_2} = \frac{1}{T_{2,bulk}} + \frac{1}{T_{2,surface}} + \frac{1}{T_{2,diffusion}} = \frac{1}{T_{2,bulk}} + \rho_2 \frac{S}{V} + D_0 \frac{(\gamma G t_e)^2}{12} \quad (8)$$

Where:

ρ_2 : surface relaxivity

S/V: surface to volume ratio

D_0 : self-diffusion coefficient of the fluid

G: magnetic field gradient

γ : gyromagnetic

t_e : time between NMR spin echoes.

Because in single phase T₂ measurement, the surface relaxation time is much faster than the bulk and diffusion relaxations, it is assumed that the T₂ measurement is directly proportional to the surface relaxation decay, which can be linked to the size of the pores as given by equation 8, where the relaxation of bulk and diffusion is neglected. (Kenyon et al, 1989; Prammar, 1994; Lu et al, 2021).

$$\frac{1}{T_2} \cong \frac{1}{T_{2,surface}} = \rho_2 \frac{S}{V} \cong \rho_2 \frac{1}{r} \quad (9)$$

NMR T₂-T₂ measurements emerge as a useful tool to characterize the fluid movements between the different pore bodies. In such measurements, to ensure the repeatability of the experiments, factors affecting the measurements such as the fluids and rock properties should be controlled with no alteration to the original state. (Washburn & Callaghan, 2006)

The measurements are obtained by first inducing a radioactive 90° frequency to stimulate the protons and orient them toward the x-y plane. After that, CPMG (Carr-Purcell-Meiboom-Gill) 180° train is applied to reduce the uncertainty associated with the inhomogeneity of the magnetic field. Then, mixing time is established where the magnetization with an orientation along the z-axis is maintained where it is affected solely by T₁ relaxation. The magnetization is then reverted back to the x-y plane where a second CPMG radio 180° train is induced to acquire the measurement. As illustrated by Figure 31, 90° radio frequency field is induced followed by a train of 180° RF. The signal that captures the echo is measured after each of the 180° pulses. (Flaum & Hirasaki, 2003; Appel et al., 2011; d'Eurydice & Montrazi, 2016).

As illustrated in Figure 32, In T₂-T₂ measurements of two distinctive pore systems, at an early storage time, signals present the initial position of protons and are aligned with the diagonal line, indicating the distribution of the pore structure. At a later storage time, off-diagonal peaks might emerge with the movement of the initial signal peaks across the diagonal line, indicating the strength and direction of the exchange between the two pore systems. The absence of off diagonal peaks can be attributed to one of three cases. First is that the ratio of T₁/T₂ is too small to enable the observation of exchanges between two pore systems. The second is the absence of coupling, in which no exchange is taking place. The third is when under the presence of

extremely strong diffusional coupling, in which the exchange between the two pore bodies cannot be sampled. (Codd et al., 2011; Fleury and Soualem, 2009).

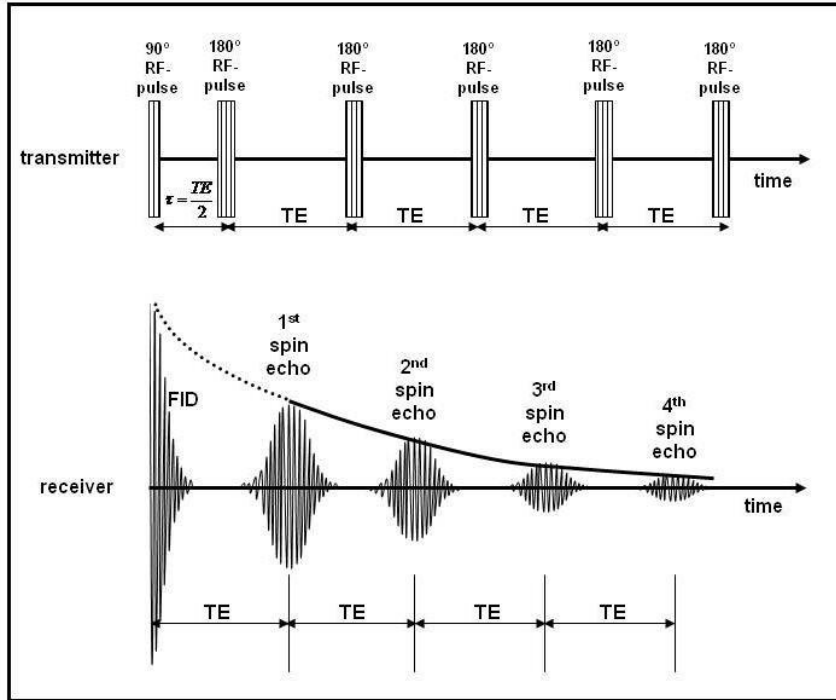


Figure 31: CPMG 1D T_2 measurement sequence, where the 2D sequence introduce storage time between each CPMG. (Appel et al., 2011; Elsayed et al., 2022).

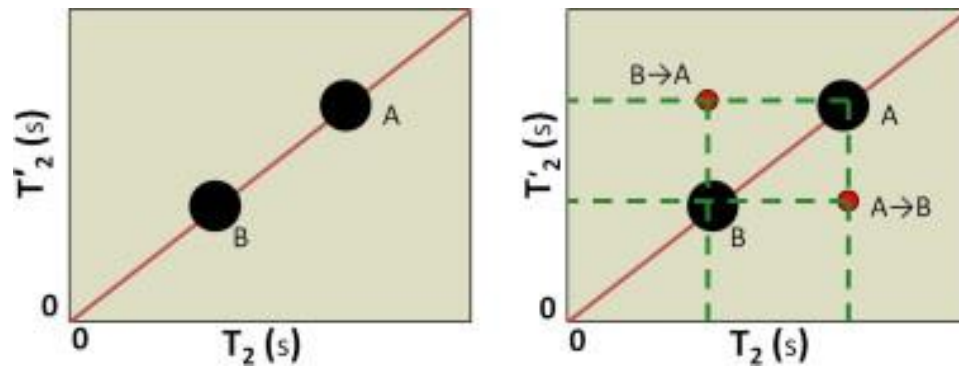


Figure 32: illustration of T_2 - T_2' signal response of two distinctive pore bodies at an early storage time (left) and late storage time (right)

T1 NMR measurements are mostly governed by the bulk and surface relaxation with an insignificant role of diffusion relaxation. T1 measurement is acquired by the pulse sequence of inversion recovery frequency (IR). Where a 180° pulse is followed by 90° , then the T1 decay is measured as illustrated in Figure 33. 2D NMR measurement refers to the event when both T1 and T2 decays are acquired. (Majumdar, 2015). When measuring T1 measurements during the relaxation acquisition time, T2 measurement can be obtained too by a extending the IR sequence to inversion recovery spin echo (IRSE) sequence, which incorporates both IR and CPMG as shown in Figure 33.

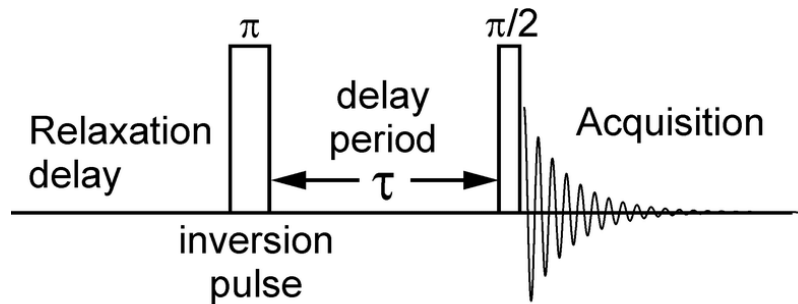


Figure 33: T1 relaxation measurement. (Majumdar, 2015)

The Pulsed-Filed Gradient (PFG), which is non also as T_2 -D, was introduced by (Stejskal & Tanner, 1965). And since then, have gained recognition in the petroleum industry as a means of investigating the degree of emulsion and examining the fluid's movements. The approach goal is to detect the kinetics of nuclei during the diffusion time which is done by introducing two magnetic field gradients. The diffusion time is defined as the time lapse between the two pulses: where the first magnetic field gradient aims at dephasing the magnetization and the second is vice versa, to reestablish the phase. Figure 34 provides an illustration of PFG sequence. (Elsayed et al, 2021; Cowan, 2015).

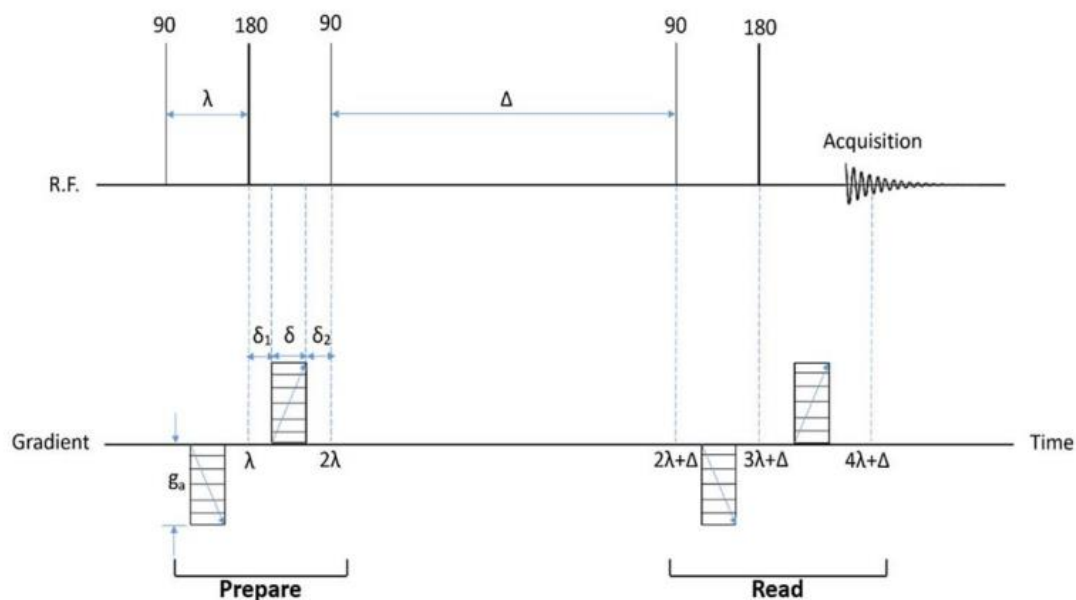


Figure 34: NMR PFG Measurement sequence. (Elsayed et al, 2021).

NMR experiments were performed using Oxford Geo-Spec2-75 2 MHz, shown in Figure 35, and Oxford Geo-Spec2-75 12 MHz. The measurements were conducted at ambient pressure and room temperature. T_2 measurements were obtained using a CPGM pulse sequence with a minimum signal to noise ratio of 100 and 0.05 ms in tau time.



Figure 35: NMR Oxford 2 MHz Geo-Spec 2, which was used in the laboratory work.

3.3 NMR Simulation

Two simulation models were developed to mimic and investigate the effect of diffusion coupling under the presence of multiple fluid systems. The developed models were inspired by the SEM imaging of the Silica Gel samples that revealed the grain distribution and some additional characteristics of the sample. Models were constructed digitally by micro-CT scanned imaging of the sample with 900^3 voxels. Simulation enables the computation of 3D maps of the internal magnetic field, then perform randomized walk simulations to generate a signal decay of CPGM pulse. (Ling et al., 2022)

Model A is a vuggy system that has a total porosity of 79% with a large spherical grain of an average size of 500 μm . The model's intergranular porosity has an average size of 45 μm . The model's pore body is divided between micro and macro systems with a ratio of 56% macro to 44% micro. Model B has a total porosity of 48%, which is more relative to the Silica gel samples, and has a similar grain size to Model B. The model's intergranular porosity averages at 18 μm . The modality ratio of Model B is 73% macro to 27% micro, which is distanced from that of Silica Gel, which is much closer to Model A. Figure 36 provides an illustration of the models' structure.

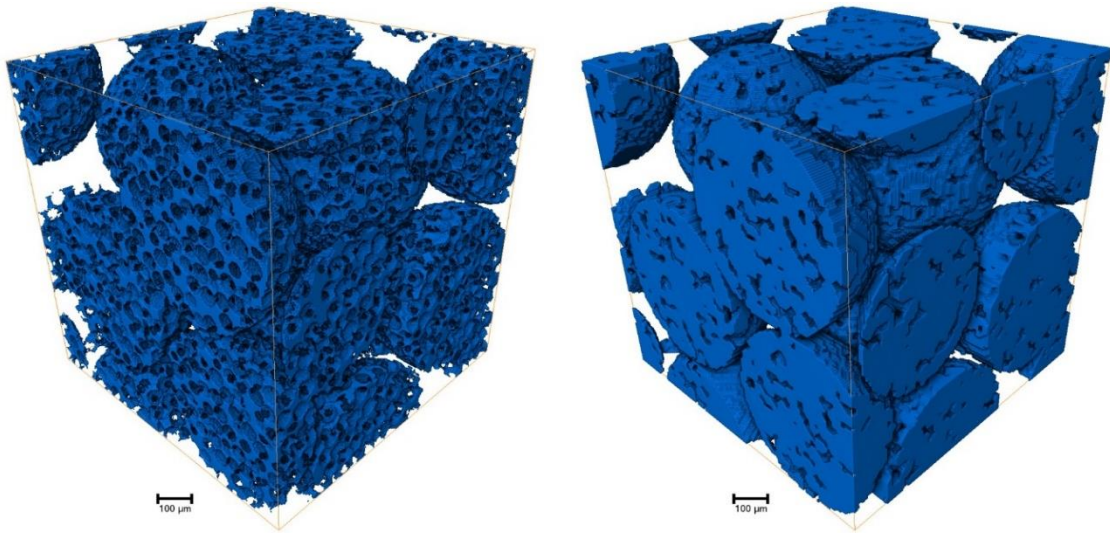


Figure 36: 3D constructions of the models used for simulation, showing in the left Model A and on the right Model B.

CHAPTER 4

Results and Discussion

The results and outcomes of this study are presented in this chapter along with their interpretations. The section includes NMR measurements of Silica Gel samples and core samples, core petrophysical measurements, fluids measurements, Mercury Injection measurements of core samples, as well as NMR simulation measurements. The primary objective of this work is to investigate the extent of fluids' properties effect on diffusion coupling. However, an understanding of interconnected factors is needed to achieve the goals of this study. As such, this section discusses the effects of pore structure, wettability, fluids bulk relaxation as well as fluids' diffusion coefficient on diffusion coupling, with a focus on the last.

4.1 Effect of Pore Structure and Grain Size

Figure 37 shows NMR relaxation of multiple Silica Gel samples with different pore and grain sizes, under full water saturation. Samples with grain sizes relatively closure to each other, which creates a more uniform pore body, are exhibiting a single peak relaxation. This suggests that the contrast between pore bodies can affect diffusion coupling. With lower contrast between smaller and bigger pore bodies, the sample has a higher potential of exhibiting diffusion coupling. When the samples are subjected to drainage until reaching S_{wi} , The T_2 signals of all samples with the consistent pore size converge. As samples are drained by filtration process with gravitational forces, fluids are primarily drained from the macro pore size while the micro pore body should maintain its fluids, driven by capillarity.

This is attributed to the contrast between capillary pressures of the two pore systems. Where it is not expected that the gravity force exceeds the threshold of micro pore capillary pressure, causing the fluid to exit. When samples with a consistent pore size are subjected to drainage, their T_2 signal converges as shown in Figure 38.

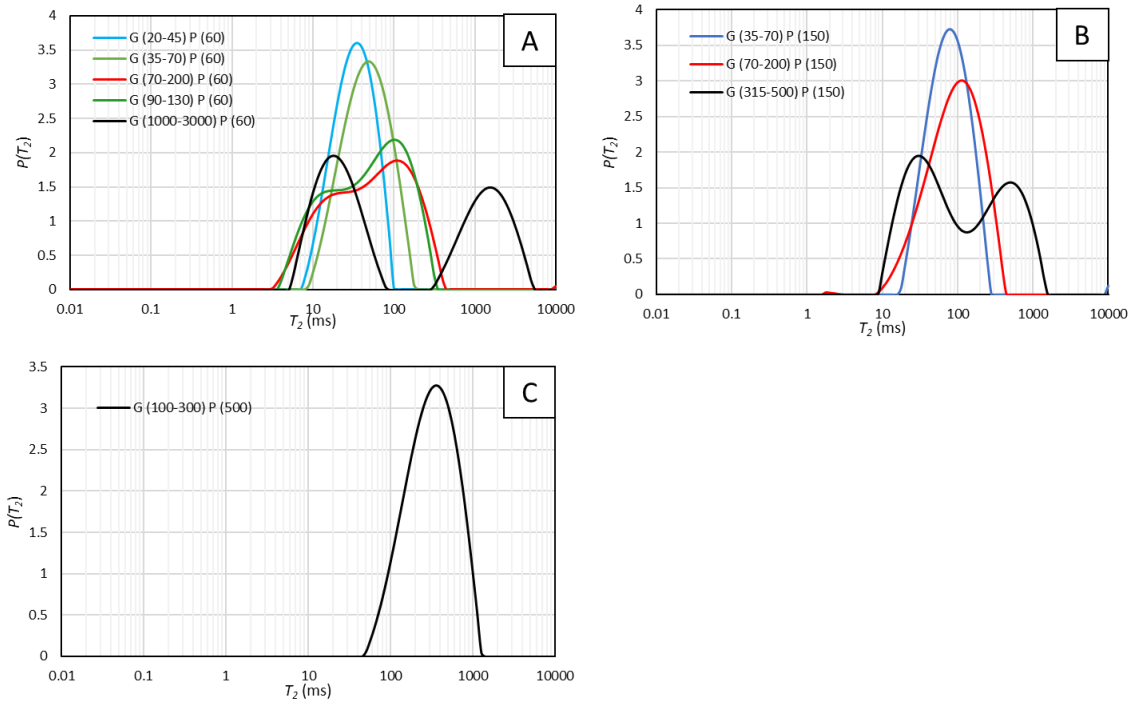


Figure 37: T_2 relaxation of Silica Gel samples that has different grain sizes and total pore sizes where: A) shows 5 samples with different grain sizes but with a pore size of 60 Å. B) shows 3 Samples with different grain sizes and a pore size of 150 Å. C) shows a sample with grain size ranges between 100 to 300 micron and pore size of 500 Å.

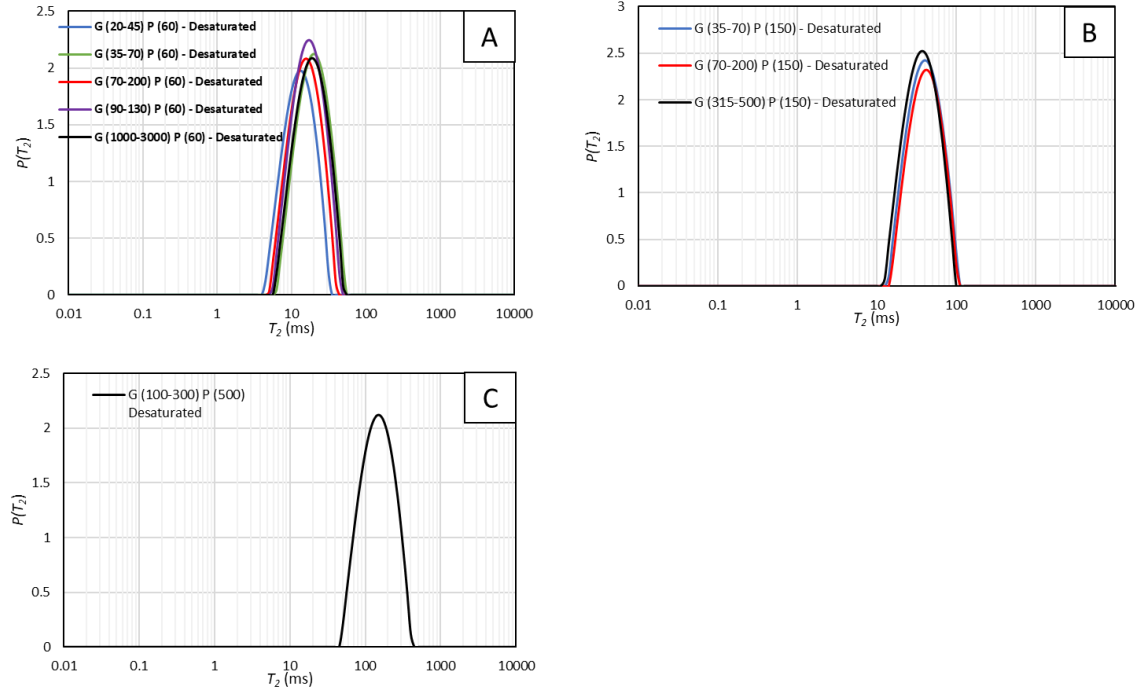


Figure 38: T₂ relaxation of Silica Gel samples that have different grain sizes and pore sizes as follows: A) 60 Å. B) 150 Å. C) 500 Å.

For fully saturated sample with a single peak, in case if the sample is not coupled, when subjected to drainage, its peak is expected to be somewhere toward the lower bound of the fully saturated peak. Thus, if the signal of drained sample is peaking lower than the boundaries of the fully saturated sample, this is a strong sign of diffusion coupling. Strong coupling can be observed from Figure 39, where drained sample shows a cumulative porosity almost half of the saturated, suggesting even distribution between micro and macro pore systems. Yet drained signal is peaking lower than the saturated sample. This means that those samples have a macro pore system of a size higher than the single peak of the saturated sample. Due to diffusion coupling a coupled peak “Pseudo average peak” between the micro and macro emerged, showing a sign of diffusion coupling.

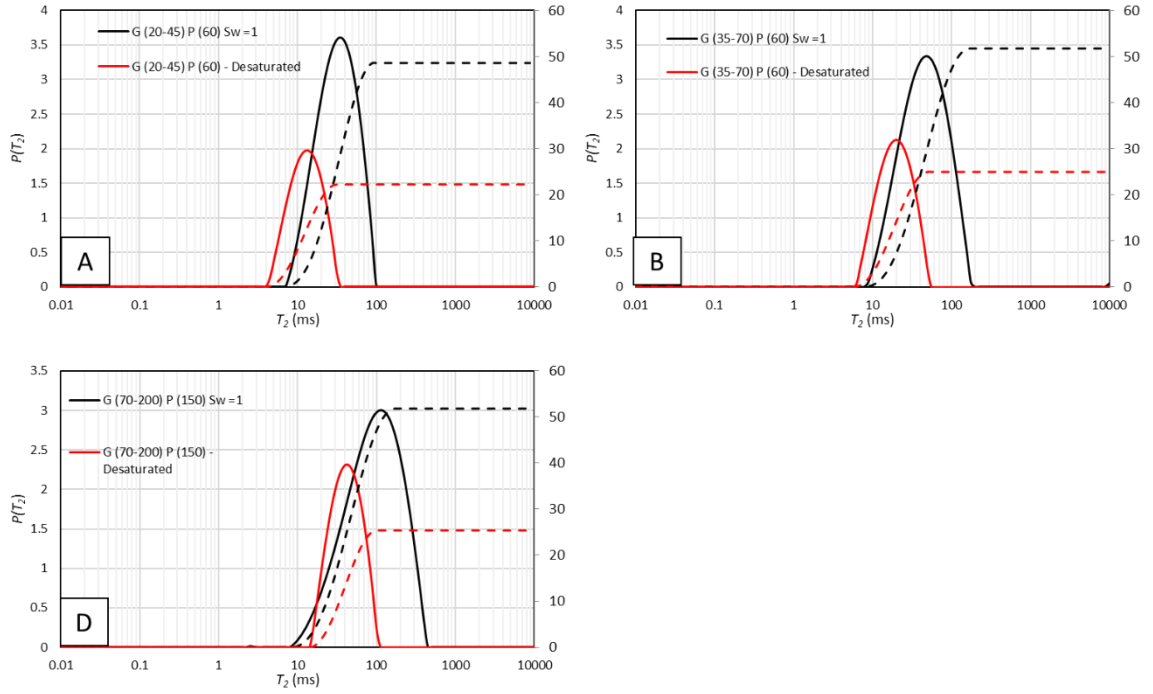


Figure 39: Examples of strongly coupled samples showing the relaxation of fully saturated and drained relaxation of samples A) G (20 - 45) P (60 Å). B) G (35 - 70) P (60 Å). C) G (70 - 200) P (150 Å).

Intermediate coupling occurs when protons are exchanging between the two pore bodies, but at a lower rates than in the strongly coupled cases. This causes the sample's relaxation to exhibit a relaxation signal with a shoulder, connecting the micro and macro systems, which can be seen in samples illustrated by Figure 40. Where the relaxation of the drained sample, which reflects the smaller pore system, captures the lower shoulder of the fully saturated sample, indicating that the sample is weakly coupled.

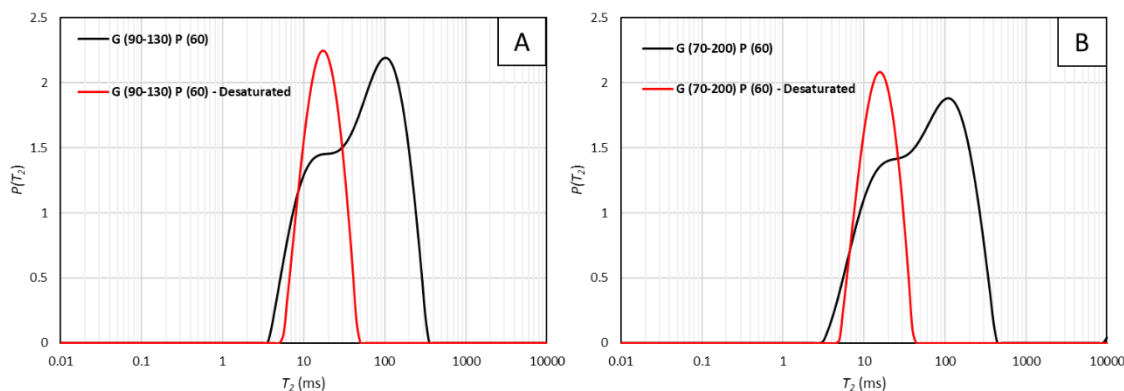


Figure 40: Examples of samples with intermediate coupling showing the relaxation of fully saturated and drained relaxation of samples A) G (90 - 130) P (60 Å). B) G (70 - 200) P (60 Å).

Lower diffusion rates between the two pore systems results in a more distinct relaxation time that has a better reflection of the sample's pore systems sizes and magnitude. Figure 41 shows examples of weakly coupled sample and a sample with no coupling. It is observed that in samples presented by Figure 40 and Figure 41.B, the amplitude of the drained sample is higher than that of the saturated sample. In the early relaxation time, due to coupling protons are able to diffuse to the larger pore size before relaxing, thus decaying at a pseudo average time. However, the drained sample relaxes faster which makes it harder for the protons to diffuse to the larger body. Table 6 shows the micro to macro pore sizes modality ratio for each Silica Gel sample, derived from saturated and drained curved.

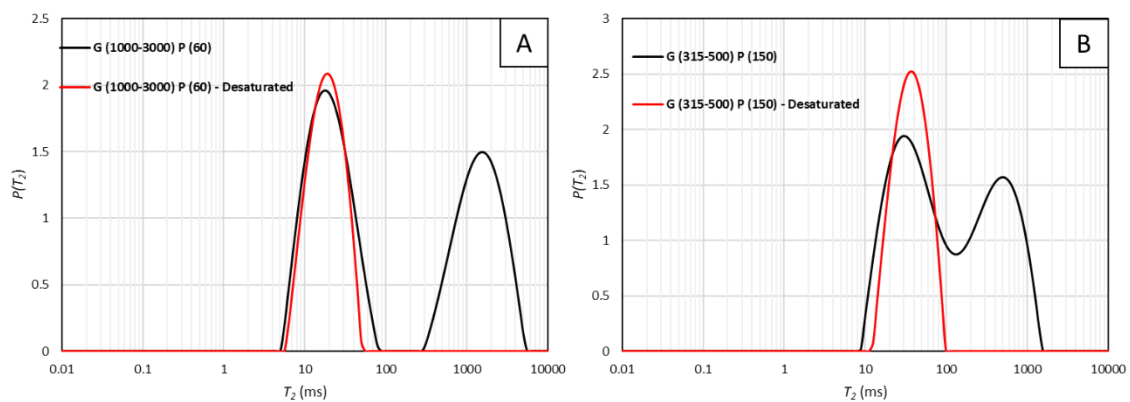


Figure 41: Examples of samples with weaker observed coupling showing the relaxation of fully saturated and drained relaxation of: A) G (1000 – 3000) P (60 Å) sample with no signs coupling. B) Weakly coupled sample G (315 – 500) P (150 Å).

Table 6: Silica Gel Samples micro and macro size distribution.

Sample label	Total porosity (%)	Smaller porosity (%)	Larger porosity (%)	Smaller/Total porosities ratio
G (100 – 300) P (500 Å)	53.48	25.61	27.86	0.48
G (315 – 500) P (150 Å)	55.42	29.05	26.37	0.52
G (70 – 200) P (150 Å)	55.07	25.36	29.72	0.46
G (35 – 70) P (150 Å)	54.34	27.82	26.53	0.51
G (1000 – 3000) P (60 Å)	50.62	24.68	25.94	0.49
G (70 – 200) P (60 Å)	51.25	23.88	27.37	0.47
G (90 – 130) P (60 Å)	54.73	26.23	28.50	0.48
G (35 – 70) P (60 Å)	51.80	24.94	26.86	0.48
G (20 - 45) P (60 Å)	48.61	22.17	26.44	0.46

4.2 Effects of Fluids' Properties

Fluids' properties are expected to have a direct effect on diffusion coupling, which is the focus of this thesis. Fluids' NMR bulk relaxation is governed by viscosity and temperature, where temperature is kept constant to eliminate its effect throughout this work. Studies have utilized NMR to characterize fluids, including fluid typing and viscosity prediction. (Winkler et. al, 2004; Chan et. al, 2004; Hirasaki et al., 2003). Figure 42 shows the free bulk relaxation of fluids used in the study. Viscosity (values of viscosity measurements of these fluids were presented in Table 2 and Table 3 in Section 4.1) is inversely proportional to relaxation. As viscosity increases, the fluid's protons tend to relax faster, which is observed in the free bulk relaxation of the fluids. Table 7 presents the diffusion coefficient of fluid samples and Figure 43 shows T₂-D. For oil samples, higher coefficients are measured with lighter density and lower viscosity. Although 15% HCl sample is an aqueous fluid with water as the base fluid, the diffusion coefficient is bigger than that of water by three folds.

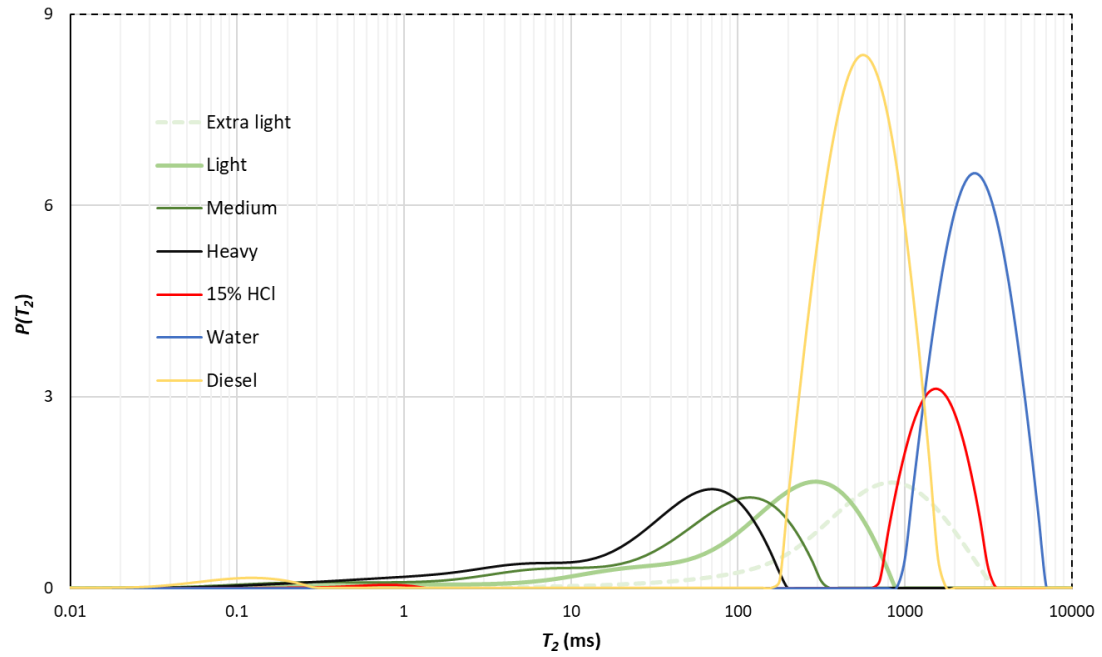


Figure 42: Free bulk relaxation of fluids utilized in the study, showing faster relaxation for more viscous fluids.

Table 7: Diffusion coefficient of fluid samples.

Label	Diffusion coefficient (m^2/s)
15% HCl	6.24×10^{-9}
Water	2.23×10^{-9}
Extra Light	1.46×10^{-9}
Arab light	1.09×10^{-9}
Medium	9.2×10^{-10}
Heavy Crude	8.51×10^{-10}

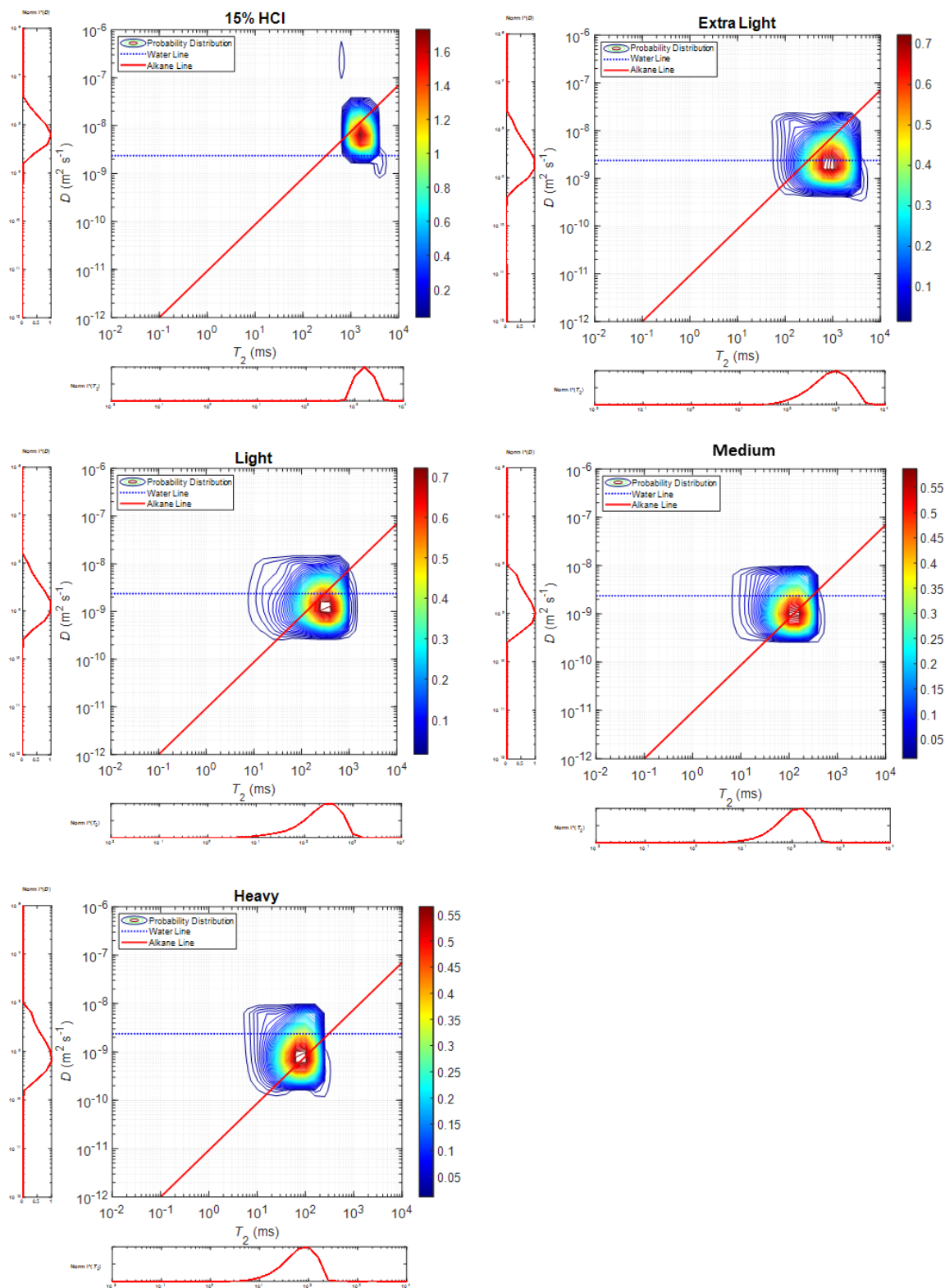


Figure 43: T2 Diffusion measurement of fluid samples.

Figure 44 shows the 15% HCl single phase T_2 relaxations of the sample G (70 – 200) P (150 Å) for both fully saturated and drained cases, while Figure 45 shows that of sample G (315 – 500) P (150 Å). Figure 45 shows that when the sample is saturated with 15% HCl, it exhibited extremely strong diffusion, as suggested by the relaxation peak time of saturated and drained cases. HCl diffusion in this sample is observed to be stronger than that of water, which showed strong diffusion. For sample G (315 – 500) P (150 Å) in Figure xx, despite that water showed no coupling, HCl showed single peak with very high amplitude. This indicates that the sample is extremely coupled. Comparing water with 15% HCl diffusion coefficient, they are within the same order of magnitude, yet 15% HCl showed extremely stronger coupling, even in cases where water showed a complete separation with two distinct separated peaks for each pore module.

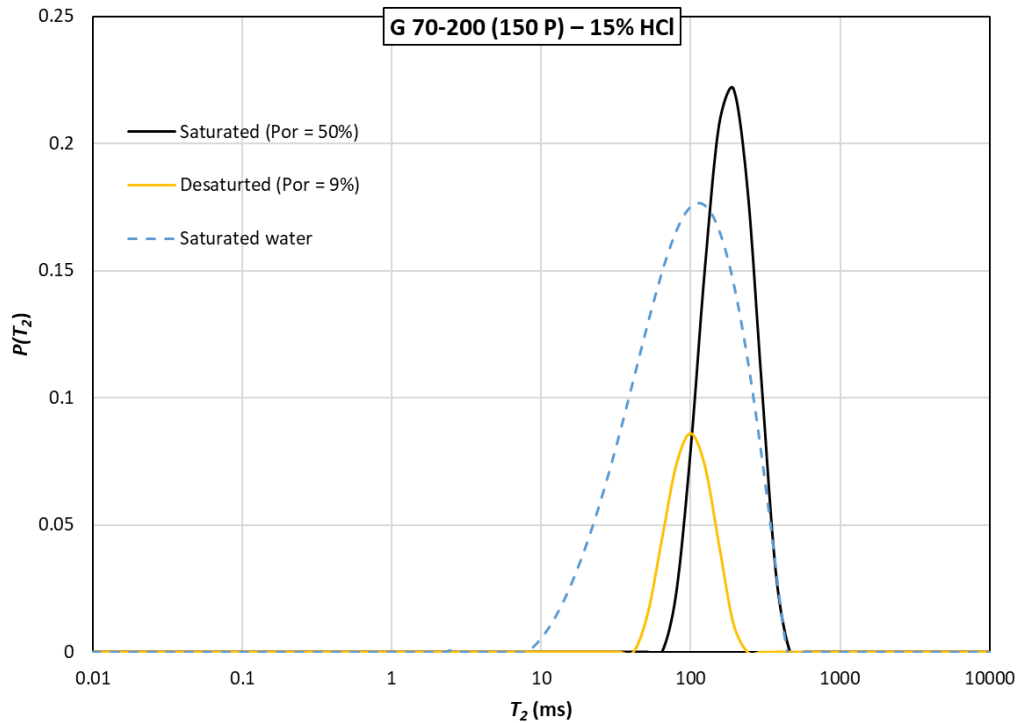


Figure 44: T_2 relaxation of sample G (70 – 200) P (150 Å) with two 15% HCl cases, saturated and drained, and compared to the relaxation of the sample when fully saturated with water.

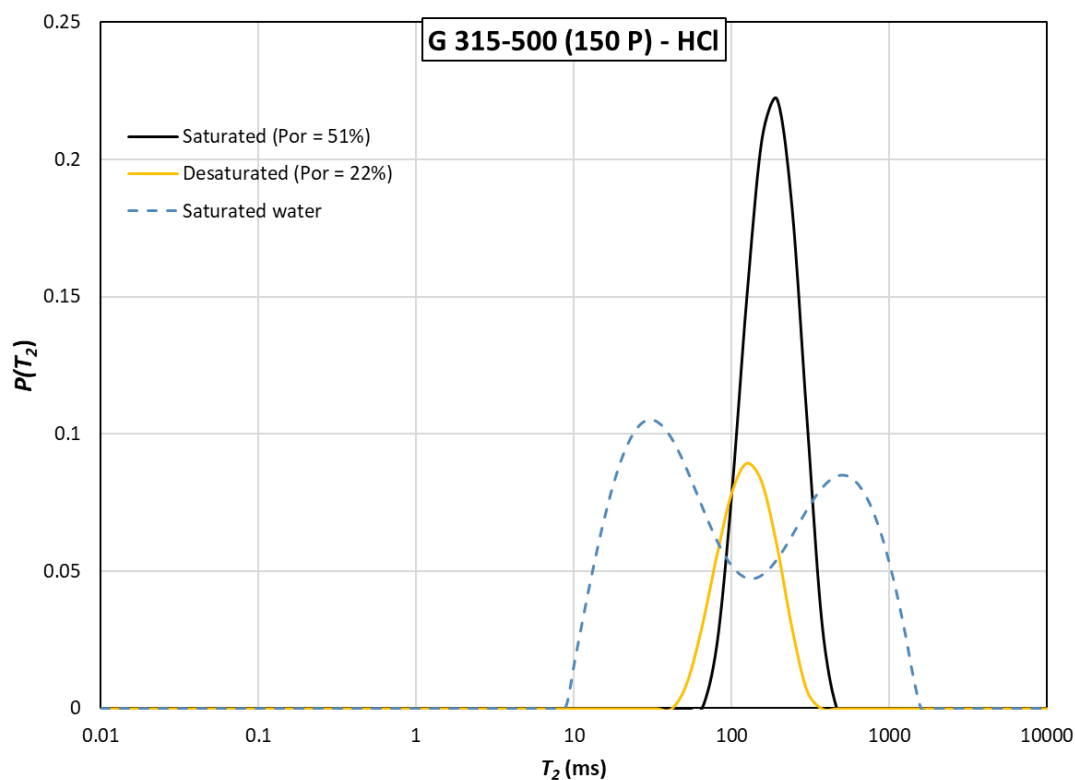


Figure 45: T₂ relaxation of sample G (315 – 500) P (150 Å) with two 15% HCl cases, saturated and drained, and compared to the relaxation of the sample when fully saturated with water.

Figure 46 & Figure 47 shows the single phase T₂ relaxations of the sample G (315 – 500) P (150 Å) for both fully saturated and drained cases, for medium crude and Arab light fluids, respectively. Figure 48 illustrates the single-phase relaxation of diesel for the same sample, which is the base fluid for oil base mud systems. Similarly, relaxation of saturated and drained cases of sample G (70 – 200) P (150 Å) are displayed in Figure 49 for medium and in Figure 50 for Arab light. The results of G (70 – 200) P (150 Å) Arab Light carried some experimental errors, as the volume of synthesized Silica Gel used in this sample was much lower than others. This results in higher plausibility of errors, evident by the NMR experimental time, where a simple T₂ measurement for this sample took more than 4 hours to reach a signal to noise ratio of 100, in comparison to few minutes for the other samples. For that, the results of this sample are considered inconclusive and are discarded. In all

four crude oil samples (Figure 46 & Figure 47 and Figure 48 & Figure 49), the saturated cases showed two peaks. The slower peak that represents the macro pore size peaked close to the fluid's free bulk relaxation peak. This is an indication of dominant bulk behavior in the macro pore system. As the sample undergoes drainage, the bulk dominant effects fade away, as shown by the desaturated cases. The diesel saturated and drained cases showed single peaks, where the drained have relaxed slower. This cannot be directly linked to coupling, as the drained sample peaked far away from saturated. This can be attributed to surface charges and adhesion of the fluid to the surface. Further studies with wettability alteration is required to evaluate the extent of effect wettability plays in this case. No conclusive conclusions can be drawn from those cases. This will not be addressed in this thesis, as it is not part of the scope of work.

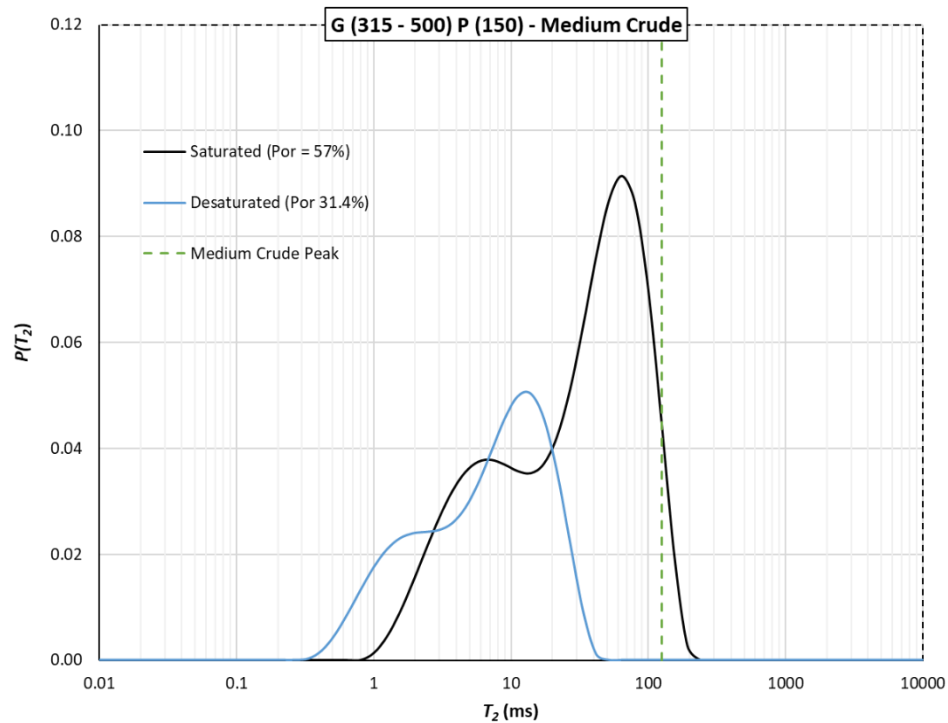


Figure 46: T2 relaxation of sample G (315 – 500) P (150 Å) with two medium crude saturation cases, saturated and drained.

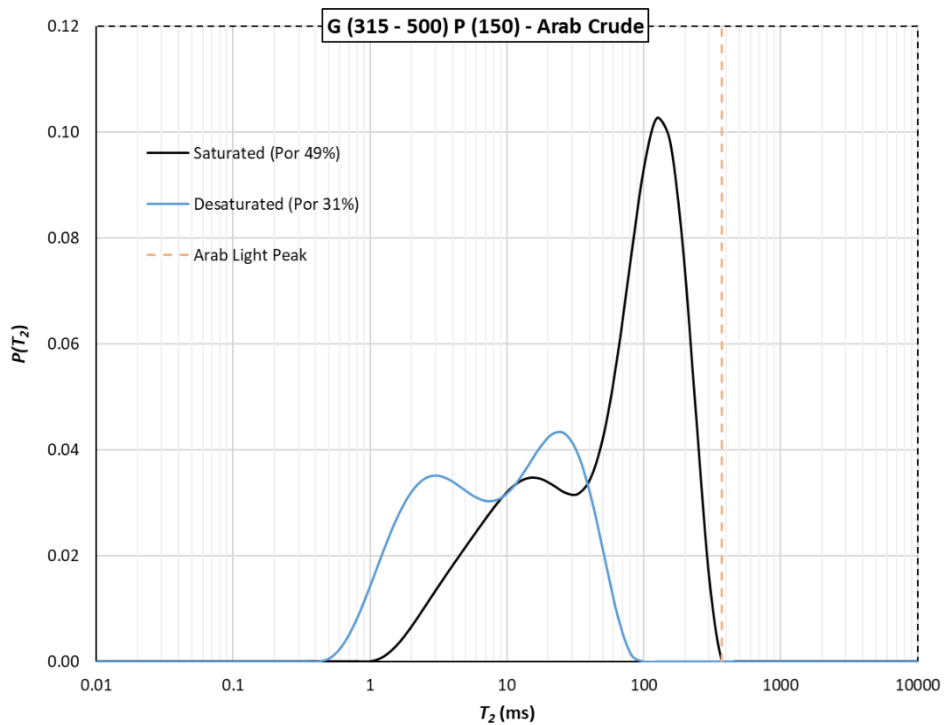


Figure 47: T2 relaxation of sample G (315 – 500) P (150 Å) with two Arab light saturation cases, saturated and drained.

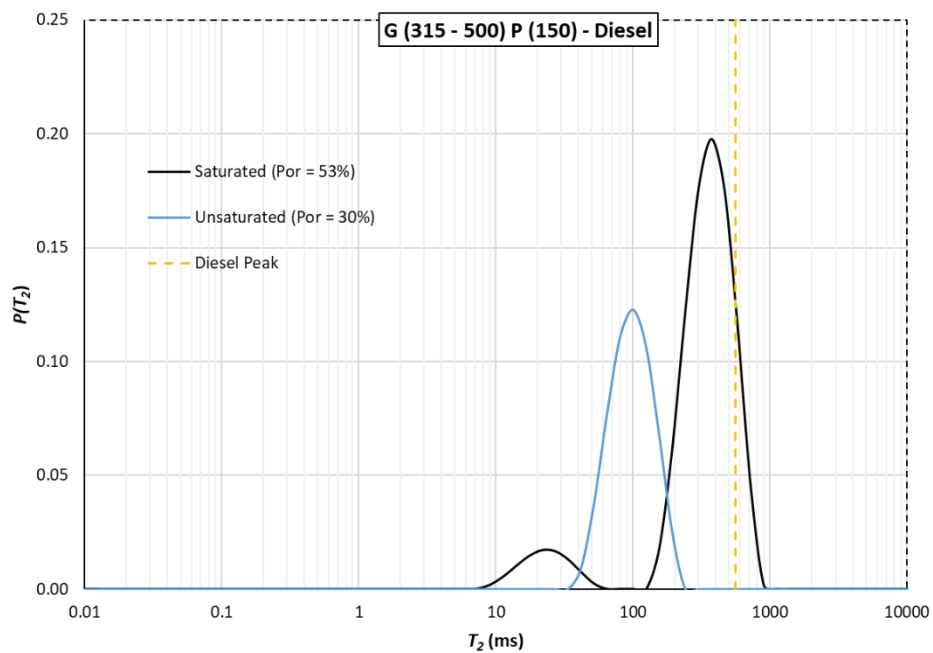


Figure 48: T2 relaxation of sample G (315 – 500) P (150 Å) with two Diesel saturation cases, saturated and drained.

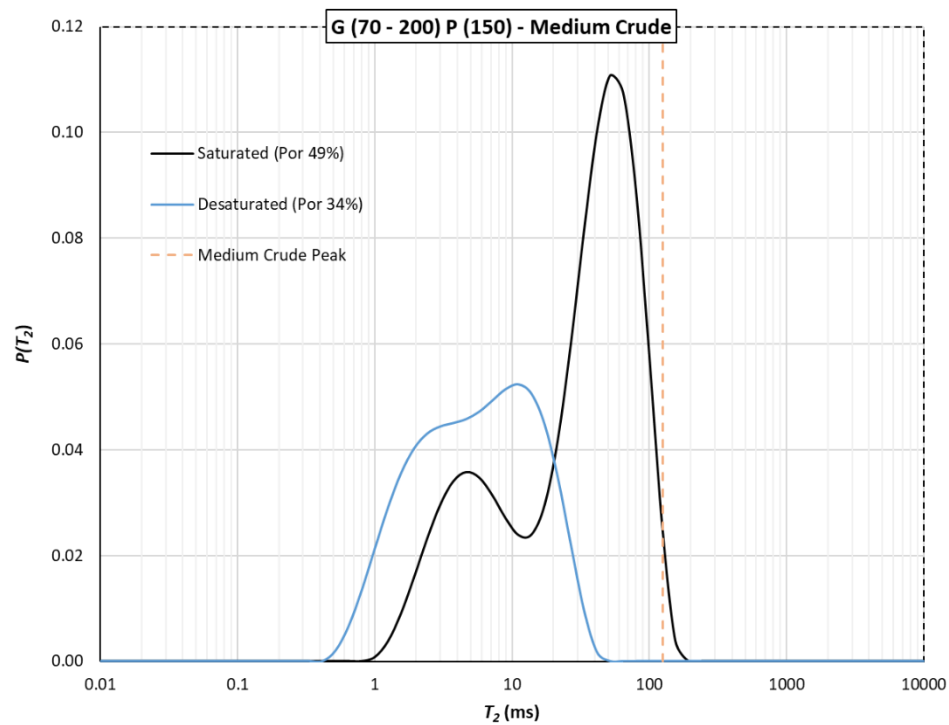


Figure 49: T_2 relaxation of sample G (70 – 200) P (150 Å) with two medium crude saturation cases, saturated and drained.

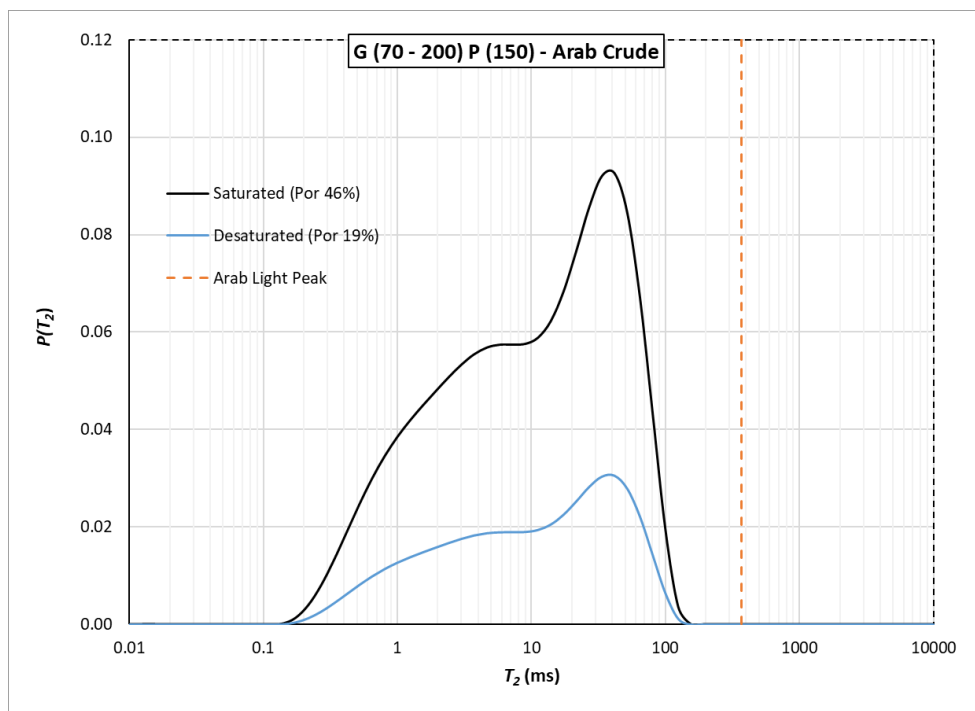


Figure 50: T_2 relaxation of sample G (70 – 200) P (150 Å) with two Arab light crude saturation cases, saturated and drained.

Figure 52 shows the 2D T_2 relaxation of sample G (315 – 500) P (150 Å) fully saturated with 15% HCl with different storage times, while Figure 51 shows medium crude oil 2D T_2 relaxation with the same sample. In support of the strong observed diffusion capacity of HCl that manifested in the 1D T_2 measurements (as discussed earlier), the 2D T_2 provides strong evidence of extreme coupling. At very early storage time, the sample showed a strong intensity at the later relaxation time, which usually reflects larger pore sizes. This relaxation behavior is consistent even when increasing the storage time. As it has been established that the porosity of the sample is equally distributed between micro and macro bodies, yet the measurement captures only the larger pore system. This gives a further explanation to the strength of HCl diffusion, where it diffuses very fast from the micro to macro bodies, in less than 1 ms. On the other hand, the medium crude 2D relaxation showed at early time a much wider intensity bubble along the diagonal line. The contour starts at very low relaxation time, which is not attributed to the pore system, but to the tail response of the fluid, which was observed in the fluid's free bulk T_2 relaxation in the beginning of this section. As the storage time increases, the contour intensity progresses along the diagonal line toward slower relaxation zone. It reaches equilibrium at 200 ms (F), where no changes are observed at later storage times (G & H). No meaningful off-diagonal counters are observed in neither of sample. The small counters present in either of samples fall far away from the origin locations of pore bodies, thus can be considered as noise. The absence of the off-diagonal peaks in HCl sample can be attributed to the strong diffusional coupling, where protons are constantly exchanging between the two pore bodies, which cannot be sampled. This is aligned with the observations made from HCl 1D measurement, which

were discussed earlier in this section. On the opposite side, their absence in the crude oil sample can be attributed to the weak coupling.

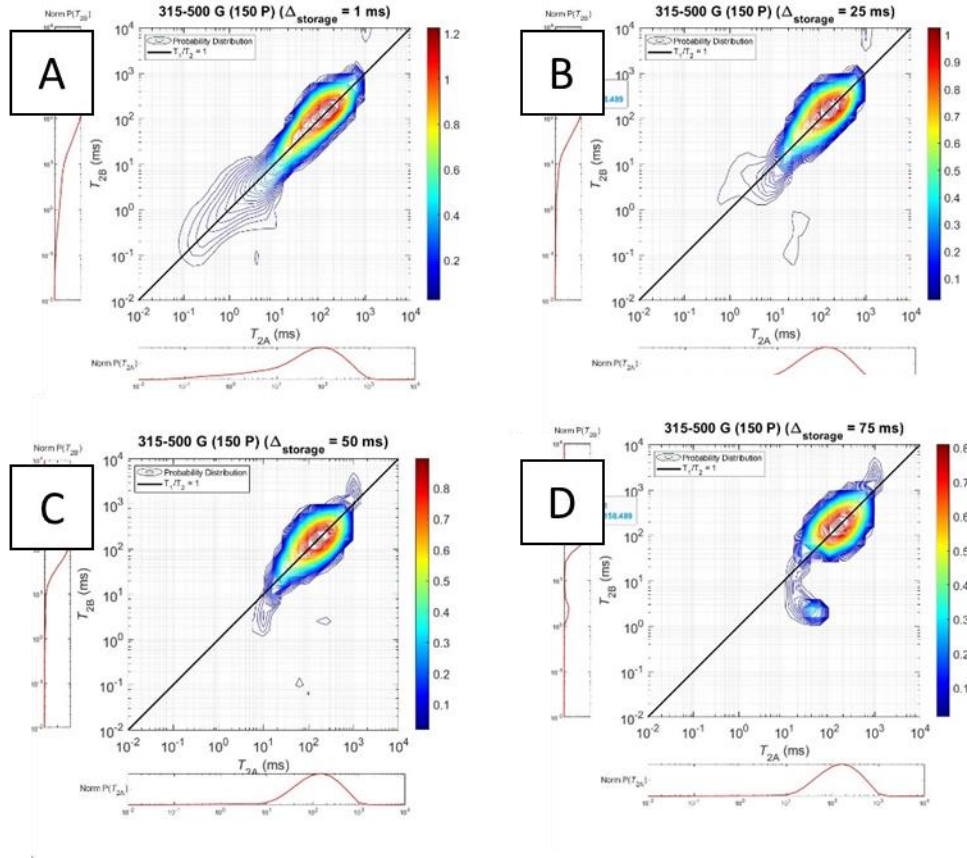


Figure 51: 2D T2 relaxation of sample G (315 – 500) P (150 Å) fully saturated with Arab medium crude under different storage time as: A) 1 ms. B) 25 ms. C) 50 ms. D) 75 ms.

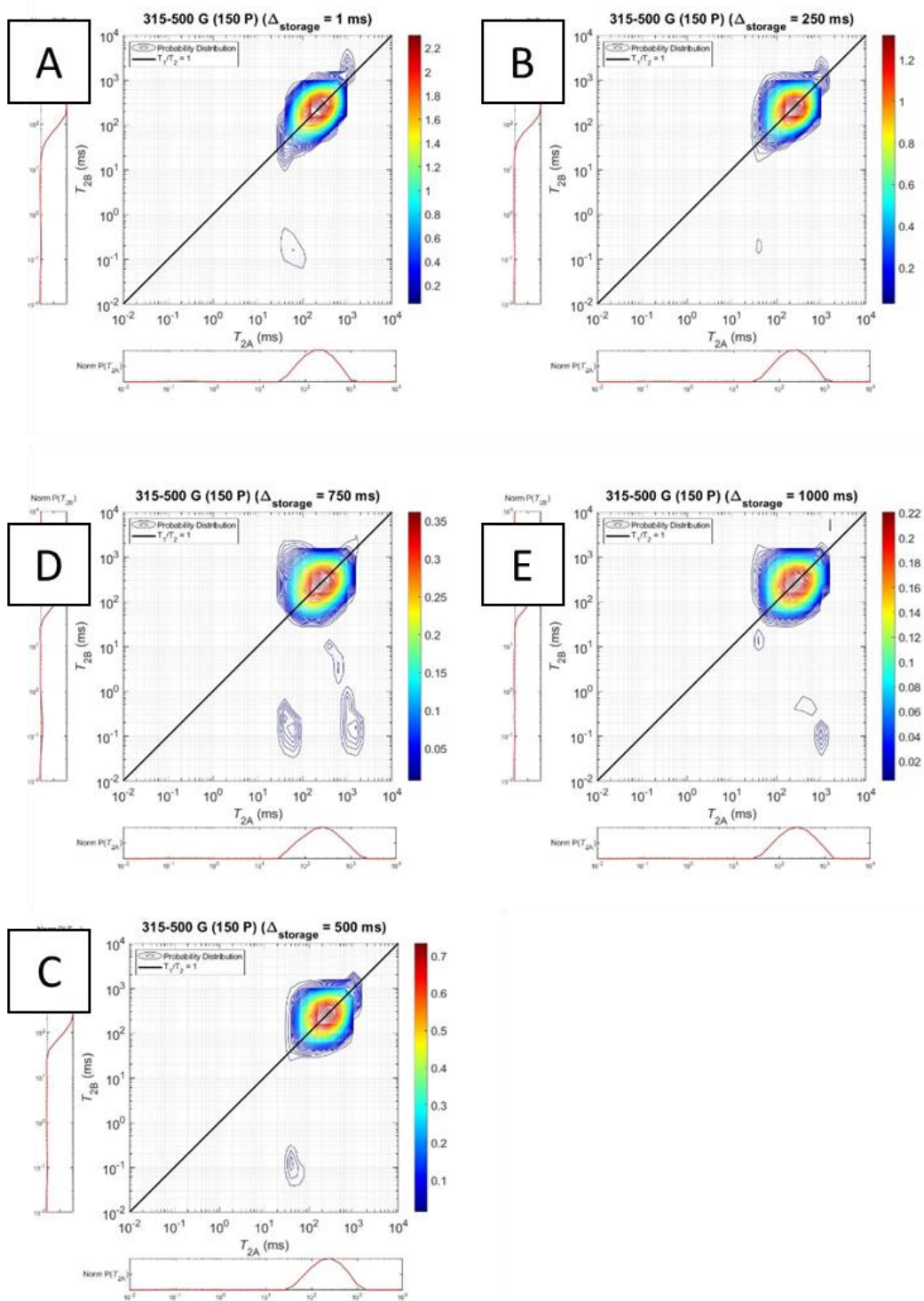


Figure 52: 2D T2 relaxation of sample G (315 – 500) P (150 Å) fully saturated with 15% HCl under different storage time as: A) 1 ms. B) 250 ms. C) 500 ms. D) 750 ms. E) 1000 ms.

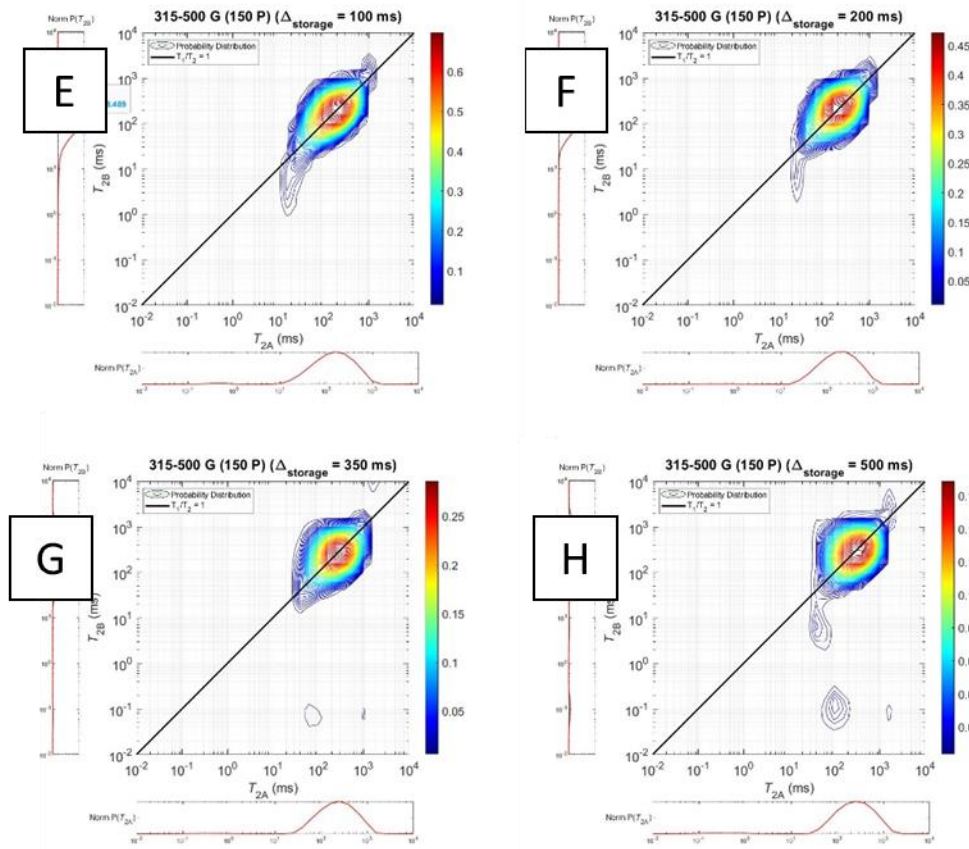


Figure 53: 2D T2 relaxation of sample G (315 – 500) P (150 Å) fully saturated with Arab medium crude under different storage time as: E) 100 ms. F) 200 ms. G) 350 ms. H) 500.

Figure 53, Figure 54 & Figure 55 shows relaxations of the samples G (60 – 200) P (60 Å) and G (60 – 200) P (150 Å), respectively, under different saturation phases, going from single phase water to S_{wi} then saturated with oil at S_{wi} . For the sample G (60– 200) P (60 Å), the sample exhibited intermediate coupling as discussed in detail earlier in this section. Remarkably, when introducing oil to the sample two peaks emerged, reflecting the two pore systems. The Macro pore peak was dominated by the bulk fluid relaxation, where it peaked at the same time as the free bulk fluid's peak. This indicates that the oil phase does not wet the system and there is no surface relaxivity effect. The amplitude of the smaller pore system is reduced at multi-phase compared to desaturated case because the signal is

dominated by the bulk relaxation of the fluid. The same trend was observed with the second sample, where originally it showed strong coupling. When injecting oil at S_{wi} it showed a complete separation between the peaks representing the two pore systems. Recall Figure 49, which show relaxation of the same sample in Figure 55 with single phase oil, the sample relaxed much faster than the fluid's bulk and showed no separation between the two pore bodies in two single-phase oil, whereas showed separation in multi-phase

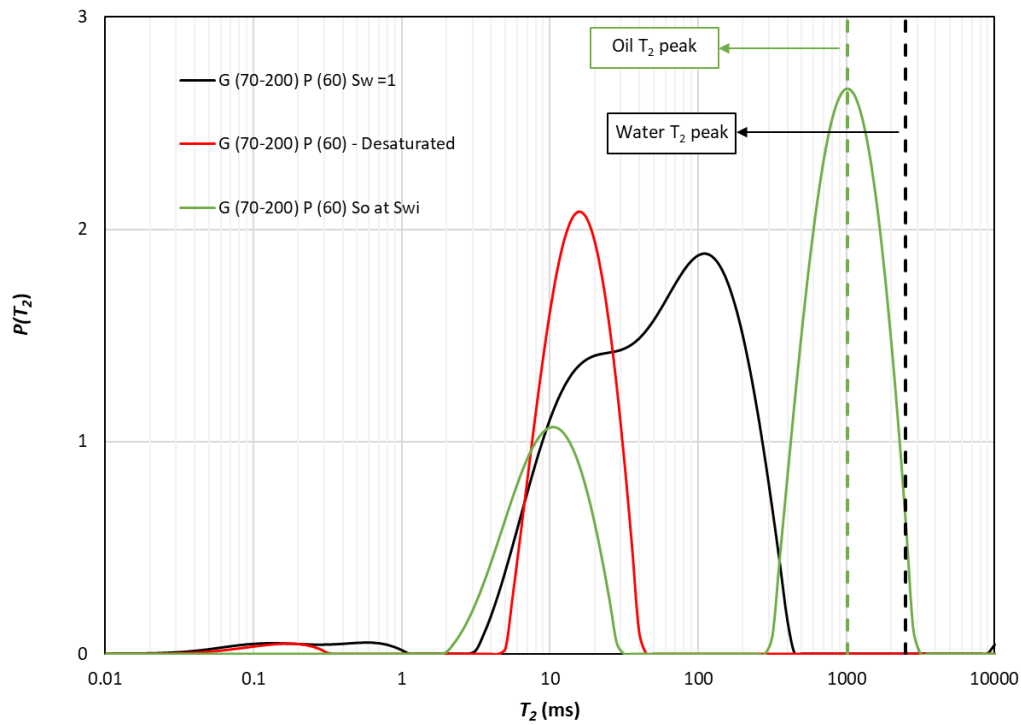


Figure 54: Relaxation of G (60– 200) P (60 Å) under multi-phase saturation, starting with fully saturated with water (black) desaturated by filtration (red) and then saturated with oil at S_{wi} (green).

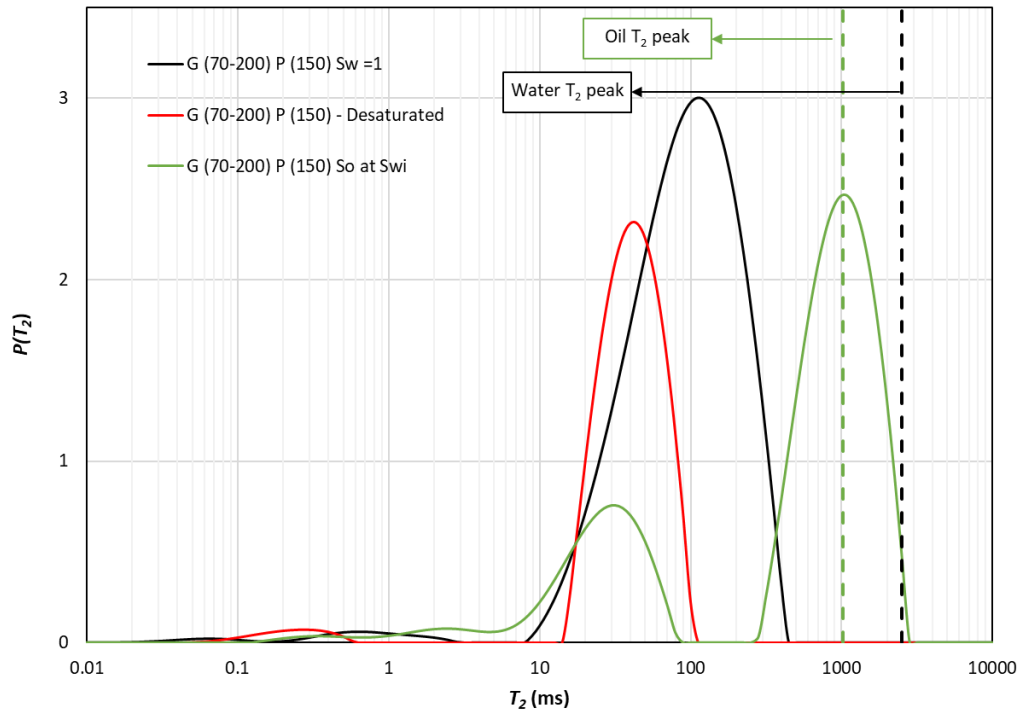


Figure 55: Relaxation of G (60– 200) P (150 Å) under multi-phase saturation, starting with fully saturated with water (black) desaturated by filtration (red) and then saturated with oil at S_{wi} (green).

4.3 NMR Simulation

NMR diffusion coupling has a high level of complexity, as it has multiple interconnected factors. There are the factors related to fluid such as diffusion coefficient and bulk free relaxation, factors related to rock structure such as pore structure, pore throat distribution and surface relaxivity, then there are factors related to fluid-rock interactions such as wettability. Those underlying factors are inherent or dependent on each other, which makes the individual evaluation of the effect extent of each element alone challenging. NMR simulation emerges as an excellent evaluation tool to investigate single factor, independently from each other, as it enables varying one parameter while keeping the others constant. In this section, fluids' diffusion coefficient and bulk relaxation where the

focus of the NMR simulation runs to have a better understanding of their effect, independent on wettability. This was done with the assumption that all simulated fluid's are strongly wetting the system.

Comparing the simulation Models A and B to Silica Gel samples, Pore size distribution plays a significant role in determining the plausibility of diffusion coupling. With a porosity modality ration of 44% micro to 56% macro, Model A has a much better representation to Silica Gel sample pore size structure, compared to a micro to macro ratio of 73% to 23% for Model B. The advantageous resemblance of model A over model B is established despite the unrealistic total porosity of 79% for model A, in comparison to a more representative values of 48% in model B. However, the simulation is governed by the structure and not the total volumes, opposite to experimental NMR where total porosity is significant. This is reflected on the simulation runs of each fluid with water, 15% HCl and crude oil, as illustrated by Figure 56 and Figure 57. Water decay in model A showed a similar respond to the water relaxation in the Silica Gel Sample G (315 – 500) P (150 Å), whereas Model B was not close to any experimental results, showing a distinct small peak for the small micro pore body. 15% HCl matched most experimental results, showing a highly coupled case. On the other hand, 15% HCl in Model B showed some separation between the two bodies. Both models produced an erroneous simulation of crude sample, dominated by it's the fluid's bulk component. This is attributed to the inability of simulation to capture the essence of crude free bulk relaxation. For this reason, in addition to using a surface relaxivity factor of wetting phase, crude simulation results did not match experimental work. Simulation offers to input only two parameters related to fluid sample, that are the peak of the fluid's free bulk relaxation and its diffusion coefficient. This is an

obvious limitation of this simulation, as the peak value of the free bulk relaxation does not capture the full relaxation behavior, where some fluids peak together, but with varying strength and speed of relaxation, resulting in different right and left endpoints of the curvatures. This is further illustrated in Figure 58, showing three different relaxations peaking at the exact same T_2 , yet each relaxation has a different degree of dispersion and amplitude. Based on the degree of resemblance of Model A to experimental cases, this model has been used to further evaluate the effect of fluid's diffusion coefficients and bulk relaxations on diffusion coupling.

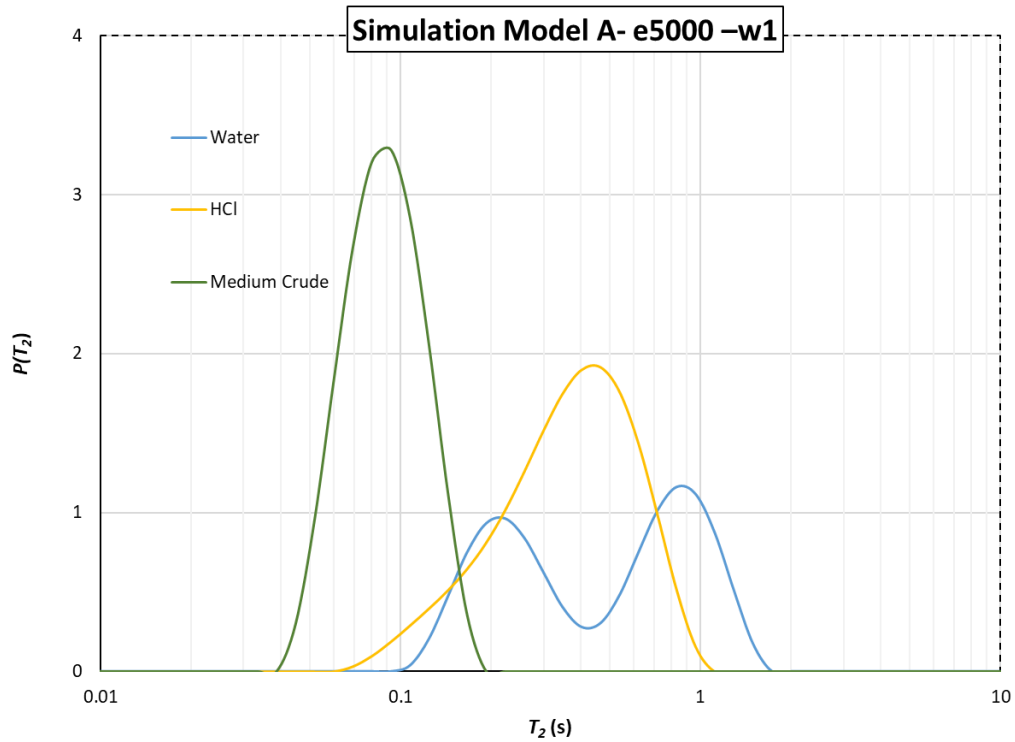


Figure 56: Three simulation runs of Model A, each run represents single-phase simulation runs saturated with 1) water. 2) 15% HCl. 3) Medium crude.

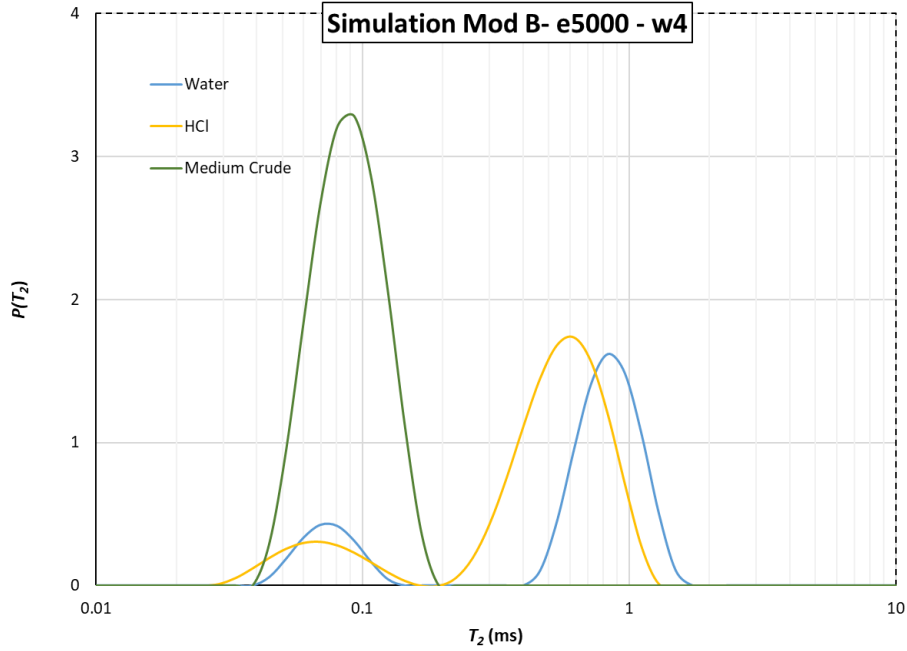


Figure 57: Three simulation runs of Model B, each run represents single-phase simulation runs saturated with 1) water. 2) 15% HCl. 3) Medium crude.

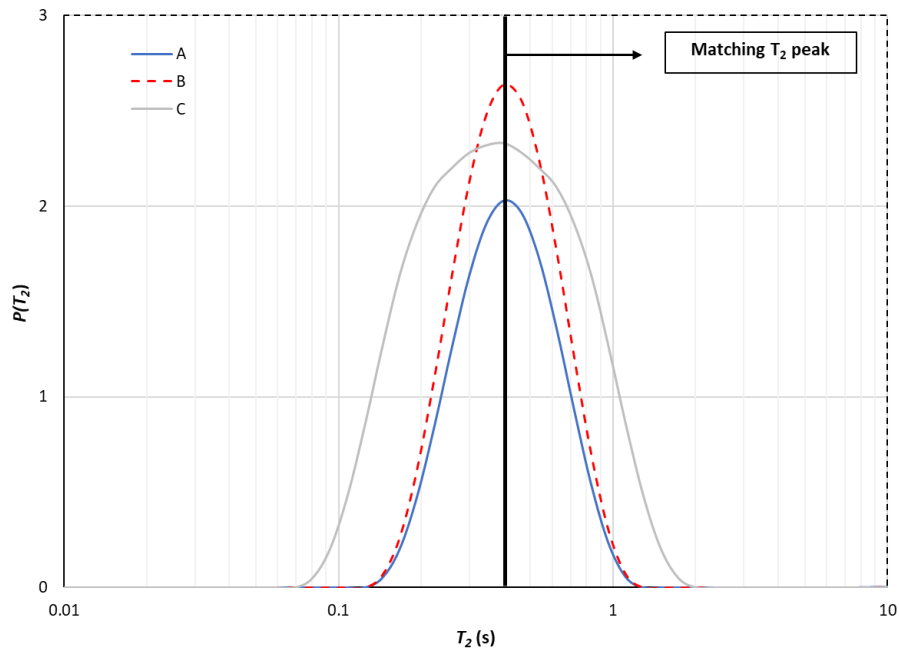


Figure 58: Illustration of the limitation of inputting to the simulation only the peak of fluids' bulk relaxation and neglecting the relaxation dispersion.

Figure 59 shows simulation run cases in which T_2 peak of the fluid's bulk relaxation is kept constant (water T_2 peak is used) while varying the diffusion coefficient. This presents a

hypothetical case that is hard to be found in experimental NMR. However, those are developed to have a controlled investigation of the fluid's coefficient on diffusion, by eliminating the effect of inherent parameters, such as bulk relaxation of the fluid, which is interconnected with diffusion coefficient. Lower diffusion coefficients produce more vivid characterization of the pore bodies. The gray solid line represents the parameter of water relaxation with diffusion coefficient of $2.23 \times 10^{-9} \text{ m}^2/\text{S}$ and T_2 bulk of 2.82 seconds. Diffusion coupling strength is observed to increase rapidly in diffusion coefficient window between $2.23 \times 10^{-9} \text{ m}^2/\text{S}$ (solid and $4 \times 10^{-9} \text{ m}^2/\text{S}$ (solid goldish line). 3.2×10^{-9} & $3.6 \times 10^{-9} \text{ m}^2/\text{S}$, which are displayed in dashed lines, present the area in transition, where 3.6 shows stronger diffusion. The transition from weak coupling (shown by $2.23 \times 10^{-9} \text{ m}^2/\text{S}$), to intermediate (shown by $2.23 \times 10^{-9} \text{ m}^2/\text{S}$), then to strongly coupled (shown by $4 \times 10^{-9} \text{ m}^2/\text{S}$) is not linear. Reducing the T_2 peak from 2.82 to 1.5 showed a similar behavior of varying diffusion coefficients, with a different transition window (Figure 60). At $T_2 = 1.5$ seconds, the transition to strongly coupled took place in diffusion coefficient window between 9.2 and $22.3 \times 10^{-10} \text{ m}^2/\text{S}$. Coefficient of $2.23 \times 10^{-9} \text{ m}^2/\text{S}$ caused a strong coupling compared to weak coupling at 1.5 and 2.23 seconds T_2 peaks, respectively. Both cases showed a relatively similar range of relaxation endpoints (T_2 at which curves builds and ends).

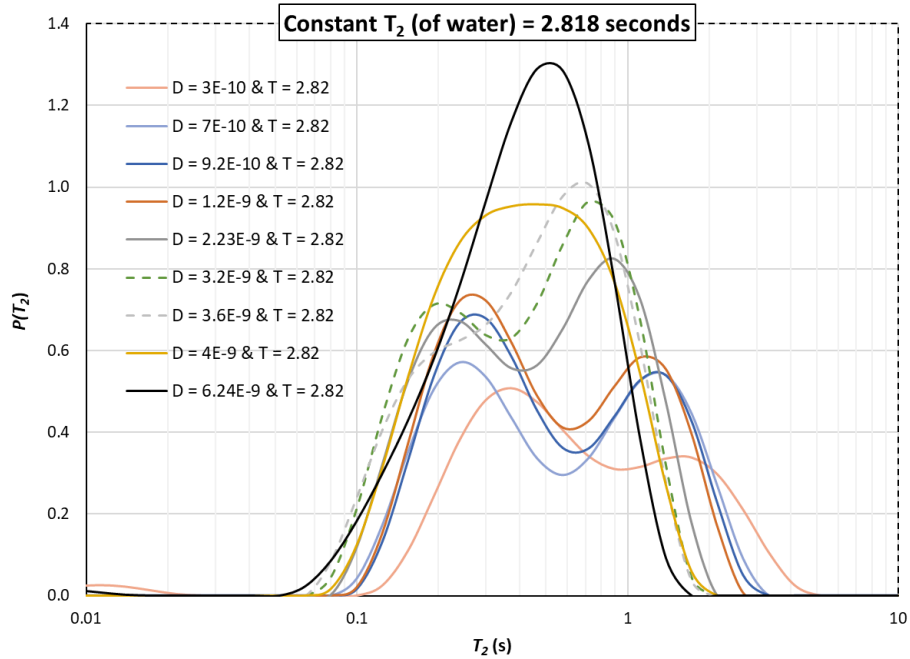


Figure 59: NMR simulations of Model A, showing 9 simulation cases with a constant T_2 peak (of water at 2.82 seconds) with varying diffusion coefficient values going from a lower end of 3 to a higher coefficient representing 15% HCl case of 62.4 ($\times 10^{-10}$ m²/S).

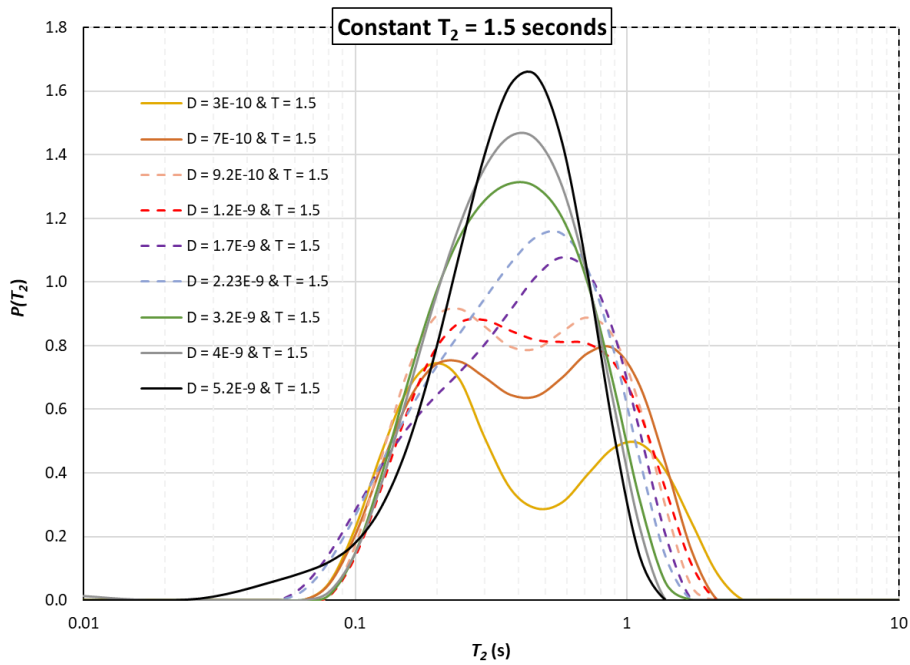


Figure 60: NMR simulations of Model A, showing 9 simulation cases with a constant T_2 peak (at 1.5 seconds) with varying diffusion coefficient values going from a lower end of 3 to a higher coefficient of 62.4 ($\times 10^{-10}$ m²/S).

Figure 61 shows the simulation results of different cases in which the diffusion coefficient kept constant at $2.23 \times 10^{-9} \text{ m}^2/\text{S}$, which represent the water value, while the T_2 values were varied. At faster fluid's bulk diffusion, the sample exhibits a coupled behavior. As the T_2 slows down, more characteristic is captured with weaker diffusion (as shown by Figure 61.B, which shows the slower T_2 peak displayed with different scale for clearer visualization.). The same trend is observed when evaluating the diffusion coefficient of 15% HCl with varying T_2 peaks. Yet, the values of T_2 at which the sample started to decouple is extremely slower than that of water, as illustrated in Figure 62). The sample showed lower coupling at a T_2 peak window between 4 and 8. The sample showed intermediate coupling at very slow fluid bulk relaxation peak at 12 seconds. Such slow values of fluid bulk relaxation cannot be found in liquids and might be even rare to find in gaseous. However, those offer a hypothetical scenario in which the extent of fluid's bulk relaxation effect on NMR diffusion can be investigated. Based on the presented simulation scenarios, the followings are observed:

- Diffusion coupling is affected by fluid's diffusion coefficient, where higher values can lead to stronger coupling.
- The relationship between diffusion coefficient and diffusion coupling is not linear. For each fluid's T_2 peak, it appears that there is a threshold after which the coupling strengths increase aggressively till reaching strong coupling.
- With a faster fluid free bulk relaxation, the diffusion coupling occurs with lower diffusion coefficient values. Similarly, with higher diffusion coefficient values, strong coupling is achieved at slow fluid's bulk relaxation.

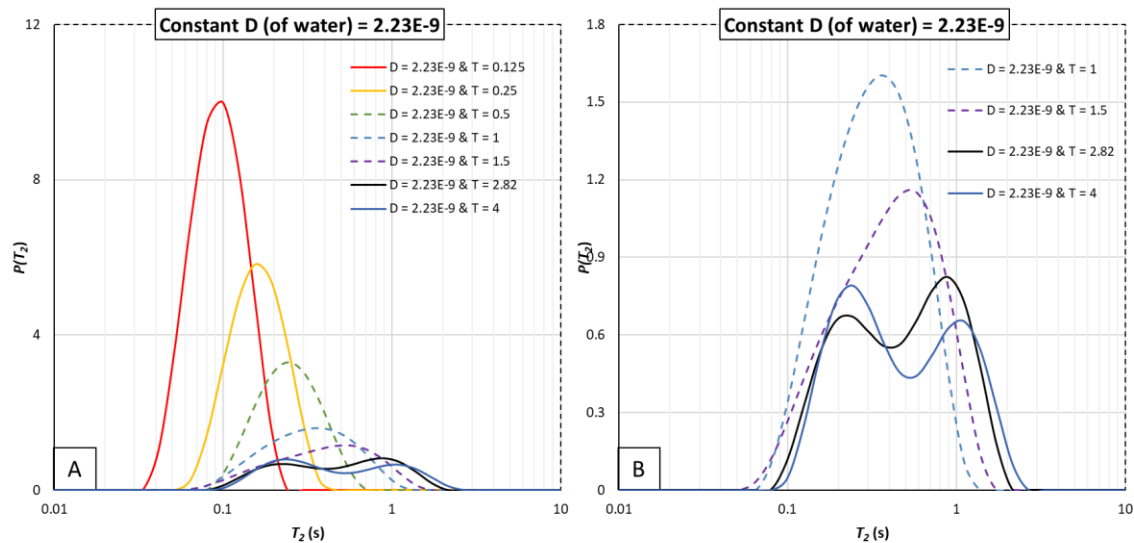


Figure 61: NMR simulations of Model A, showing 9 simulation cases with a diffusion coefficient (of water at $2.23 \times 10^{-10} \text{ m}^2/\text{s}$) with varying T_2 peaks from 0.25 seconds to 4, passing by 2.82 seconds (which represent water).

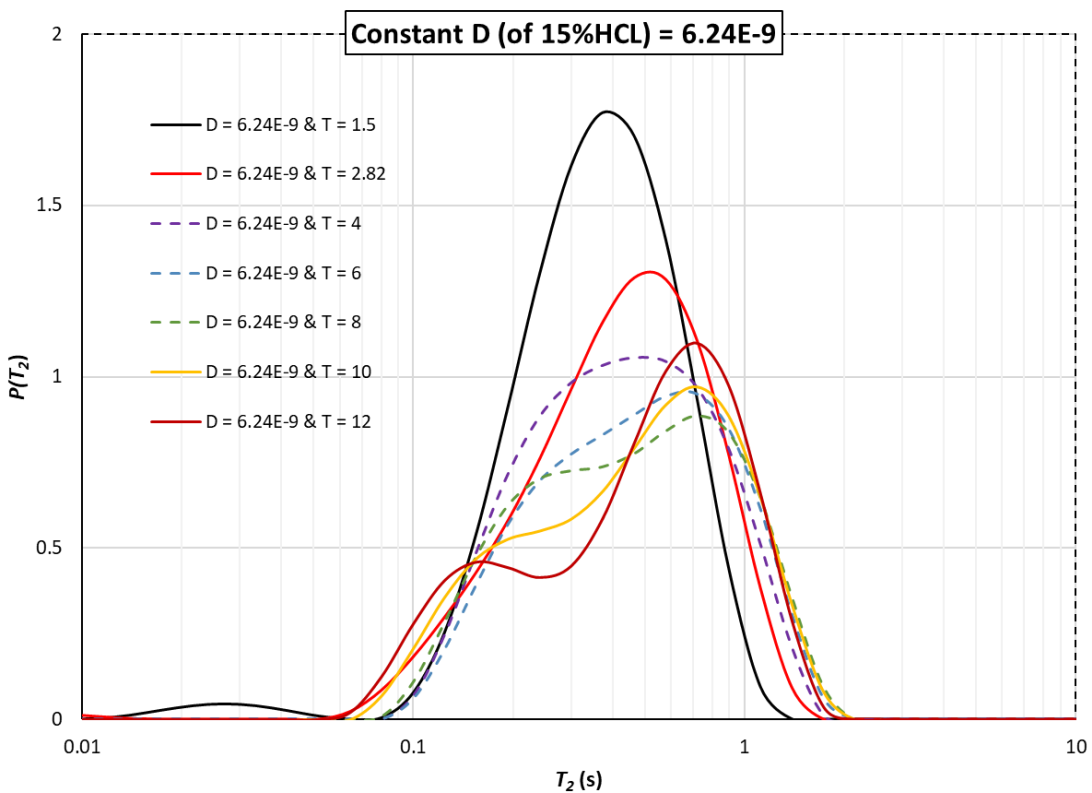


Figure 62: NMR simulations of Model A, showing 9 simulation cases with a diffusion coefficient (of 15% HCl at $6.24 \times 10^{-10} \text{ m}^2/\text{s}$) with varying T_2 peaks from 1.5 seconds to 12 seconds.

4.4 Field Core Plug Samples

This work is inspired by the operational needs to address, and ultimately, account for diffusion coupling when observed in well-logs. Due to the natural complexity of carbonate systems, this issues when encountered results in inaccurate assessment of the investigated reservoir medium. This section tests the outcomes and observations obtained from controlled medium (Silica Gel samples and Simulation) on carbonate field core plug samples. Ten carbonate rock samples were obtained and studied. The area of which the samples were obtained has a high level of diffusion susceptibility, as observed from NMR well logging. The samples' actual numbers and the field they represent were masked due to confidentiality. The samples present a wide range of petrophysics properties (permeability, pore structure, size and distribution). This can be noticed in the T_2 relaxation of samples saturated with single phase water, presented in Figure 63, Figure 64 and Figure 65. Samples diffusion coupling cannot be determined from fully saturated T_2 relaxation curve, yet there is a possibility that will be investigated later. This stems from the distinct peaks each sample relaxation is exhibiting.

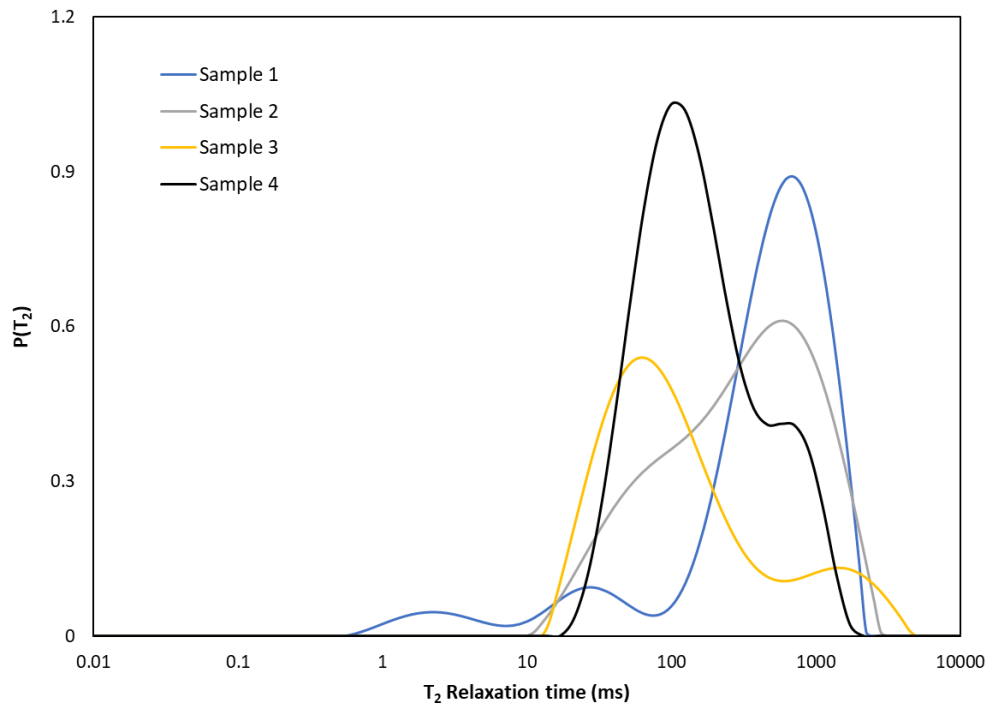


Figure 63: T₂ relaxation of core plug samples 1-4, showing different pore size and structures.

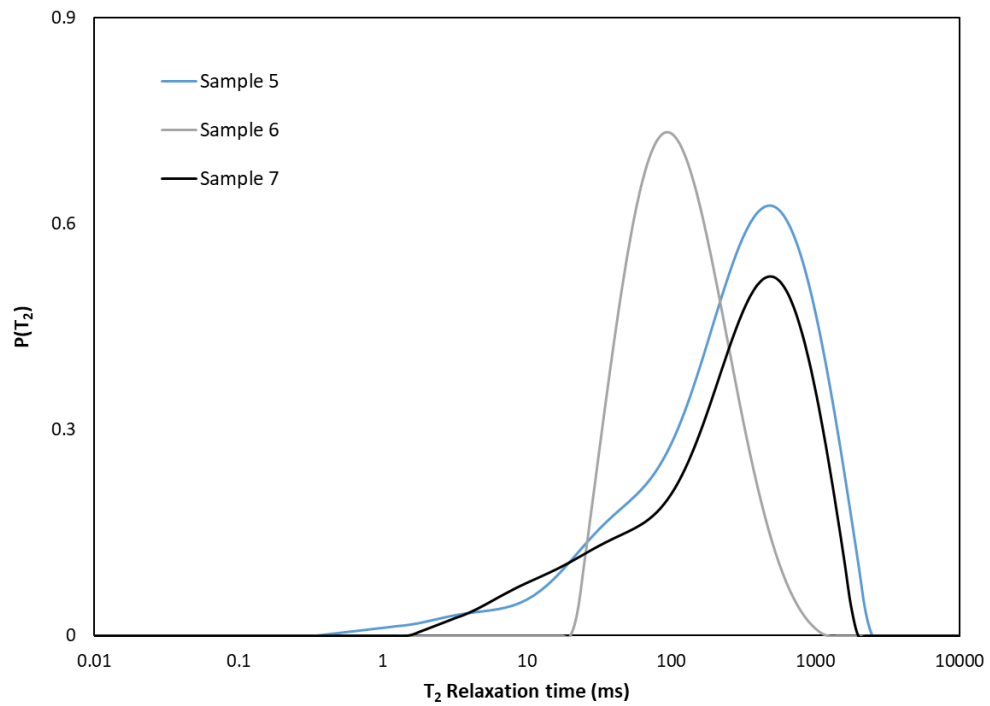


Figure 64: T₂ relaxation of core plug samples 5-7, showing different pore size and structures.

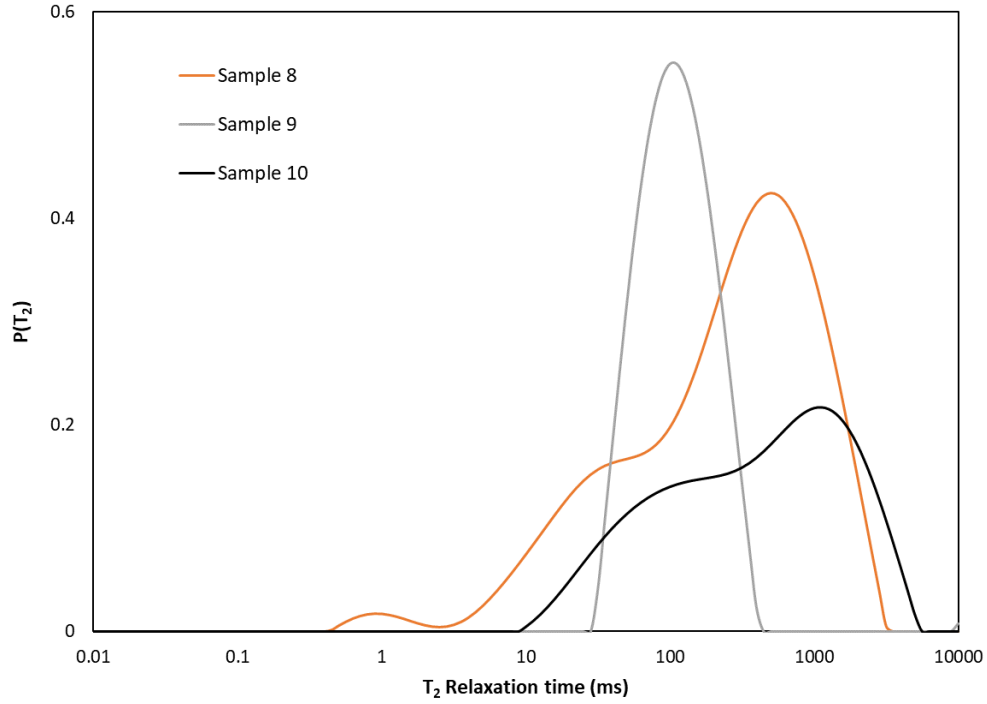


Figure 65: T₂ relaxation of core plug samples 8-10, showing different pore size and structures.

Figure 66 compares obtained petrophysical properties of the samples from NMR to the measured permeability and permeability. It shows a good match between NMR and Helium porosities. Furthermore, samples show a wide range of permeability values ranging from 0.1 to around 100 mD. Based on the samples' tightness, expressed by permeability, samples were classified into three groups. Group 1 contains samples with permeability less than 1 mD. Group 3 are samples above 10 mD, whereas samples in between those values are classified as Group 2, as shown in Figure 66.B. Both groups 2 and 3, which are the tighter samples, showed a matching trend between T_{2LM} obtained from NMR in comparison to permeability. Based on this grouping, T_2 relaxation of the samples is replotted in Figure 67, Figure 68 and Figure 69.

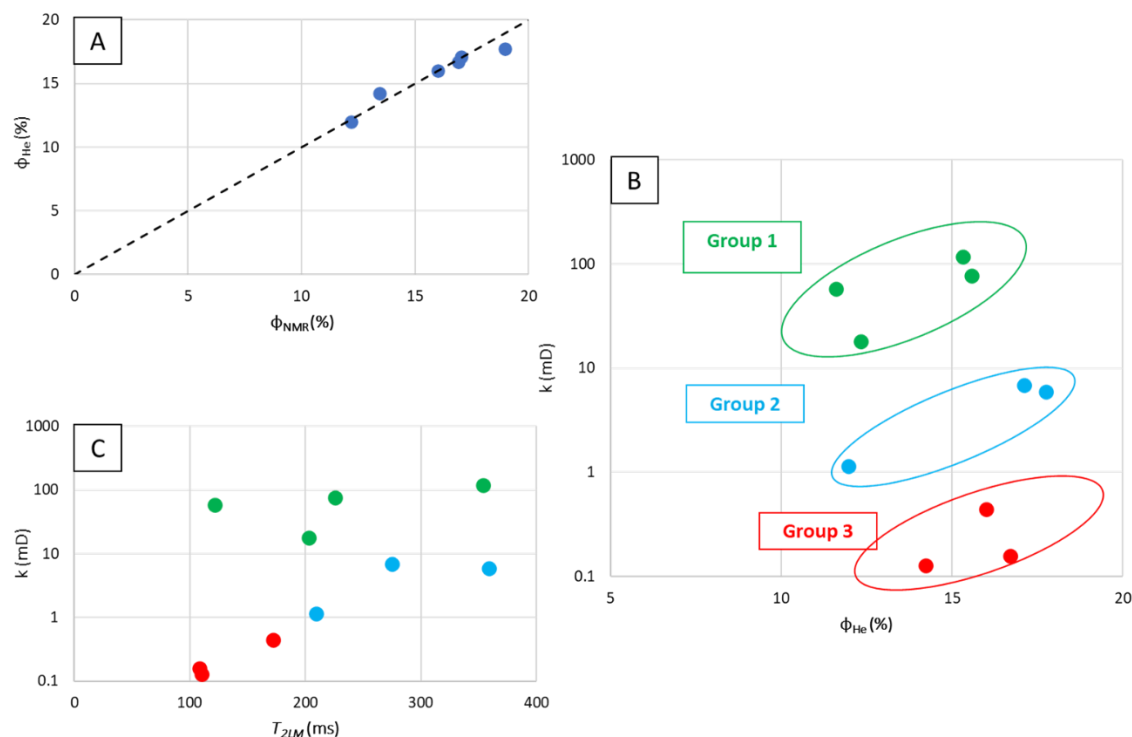


Figure 66: Basic properties of the core plug samples showing: A) comparison between Helium and NMR porosities showing a good match. B) comparison between permeability and Helium porosity, where samples were classified based on their values into three groups. C) C

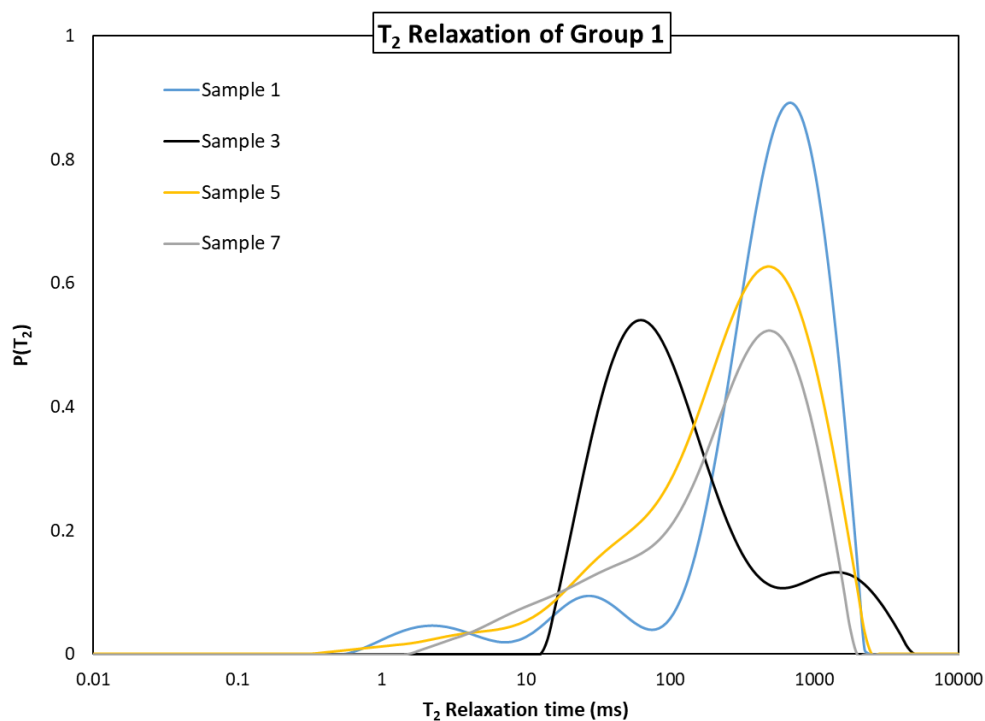


Figure 67: T₂ relaxation of core samples within Group 1

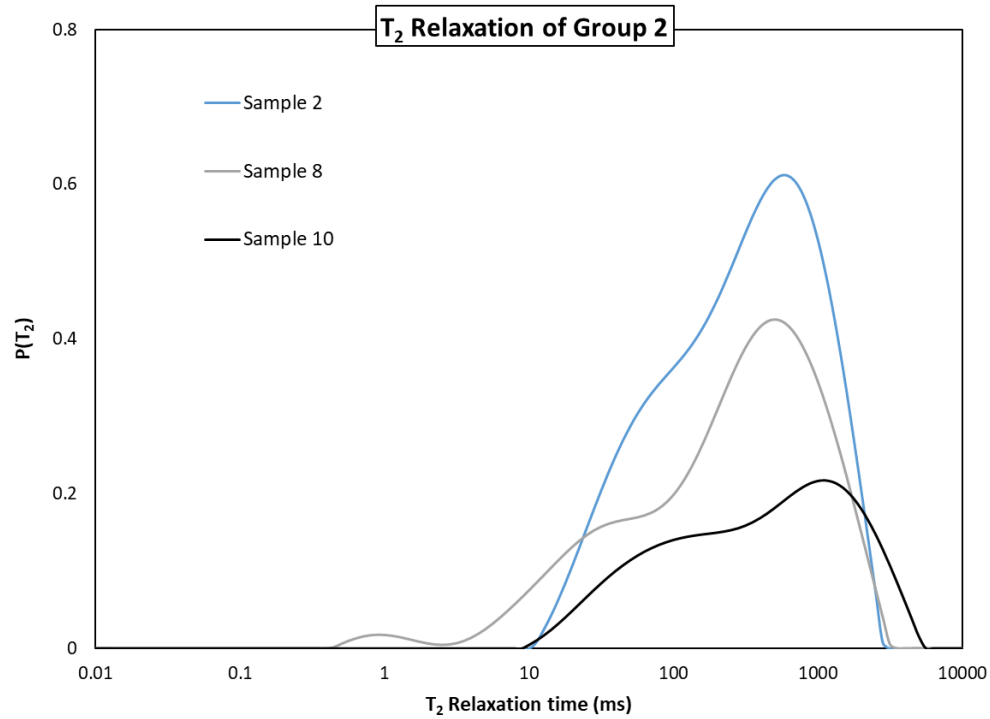


Figure 68: T₂ relaxation of core samples within Group 2

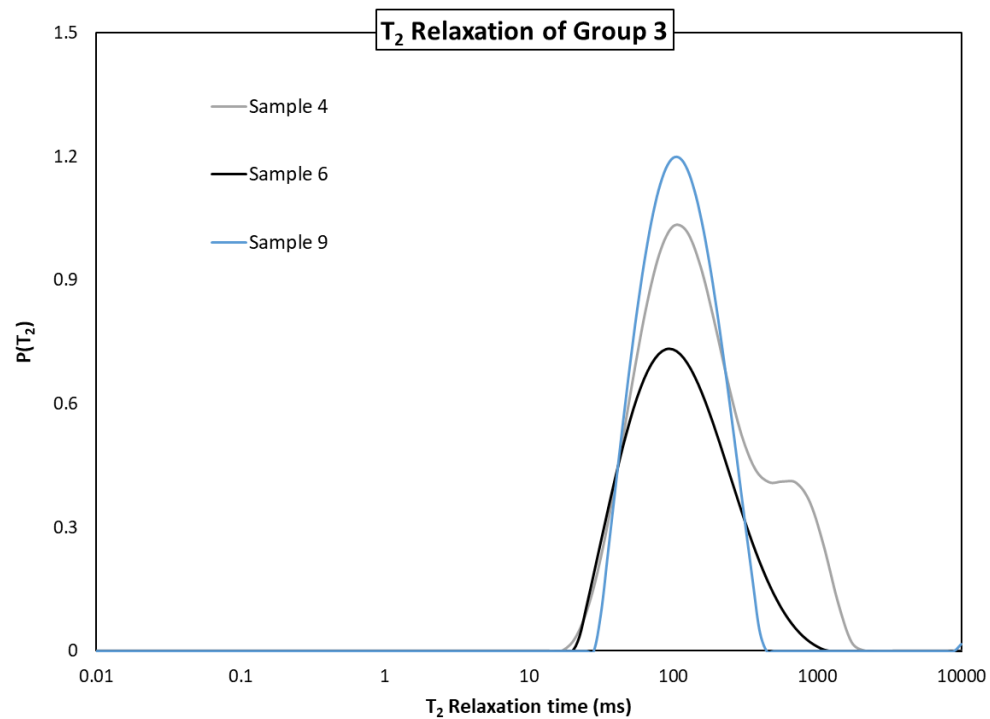


Figure 69: T₂ relaxation of core samples within Group 3

For further selection, NMR 2D & 1D T_2 measurements, MICP data (if available) along with the sample visual inspection of grain and pore size were combined to screen and pinpoint samples with high diffusion coupling potential. Sample 2 T_2 relaxation showed a single peak, indicating one pore system (Figure 70). This contradicts MICP, which shows bimodality or even trimodality (Figure 70.B), presenting a sign of diffusion coupling occurrence. Another strong evidence of this is the 2D T_2 measurement, showing a strong connectivity between two pore bodies in early storage time. As storage time progresses the protons diffuse to larger pore body (Figure 70.A). Similarly, Sample 4 showed a one connected T_2 relaxation with a shoulder (Figure 71). Yet, MICP showed two distinct pore bodies, which is supported by the 2D T_2 , where protons were located at two pore systems at early storage time, then progressed to diffuse to the larger pore between 100 and 500 ms storage time, as shown in Figure 72. From the T_2 relaxations of sample 6 and 9 (refer to Figure 69), and T_2 - T_2 measurements (Figure 72 and Figure 73), diffusion coupling determination is inconclusive. No available MICP data for those sample to further test and evaluate. Sample 10 showed a one connected T_2 relaxation (Figure 68). Yet Figure 74 showed MICP and 2D T_2 measurements, suggested two interconnected pore systems, similar to observations made on samples 4 & 6. Sample 10 has a weaker coupling compared to samples 2 & 4, as suggested by the dispersion of the sample 10 T_2 relaxation. Therefore, samples 2 & 4 were selected for further analysis.

Sample 2

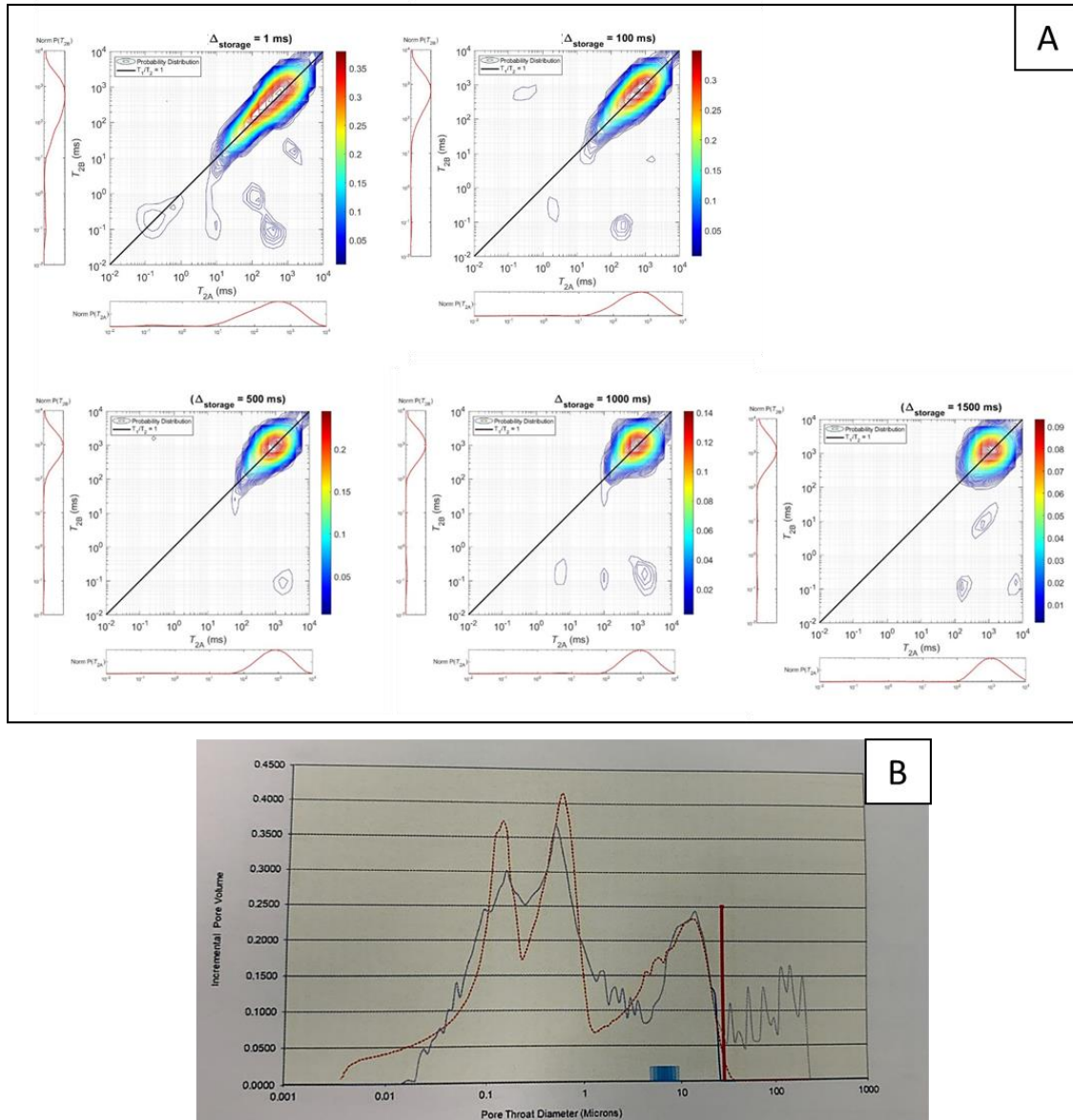


Figure 70: Sample 2 shows a high degree of diffusion coupling by: A) 2D T2 shows a strong exchange between protons. Storage times used are 1, 100, 500, 1000 and 1500 ms B) shows MICP analysis of sister sample indicating porosity bimodality, which was not captured

Sample 4

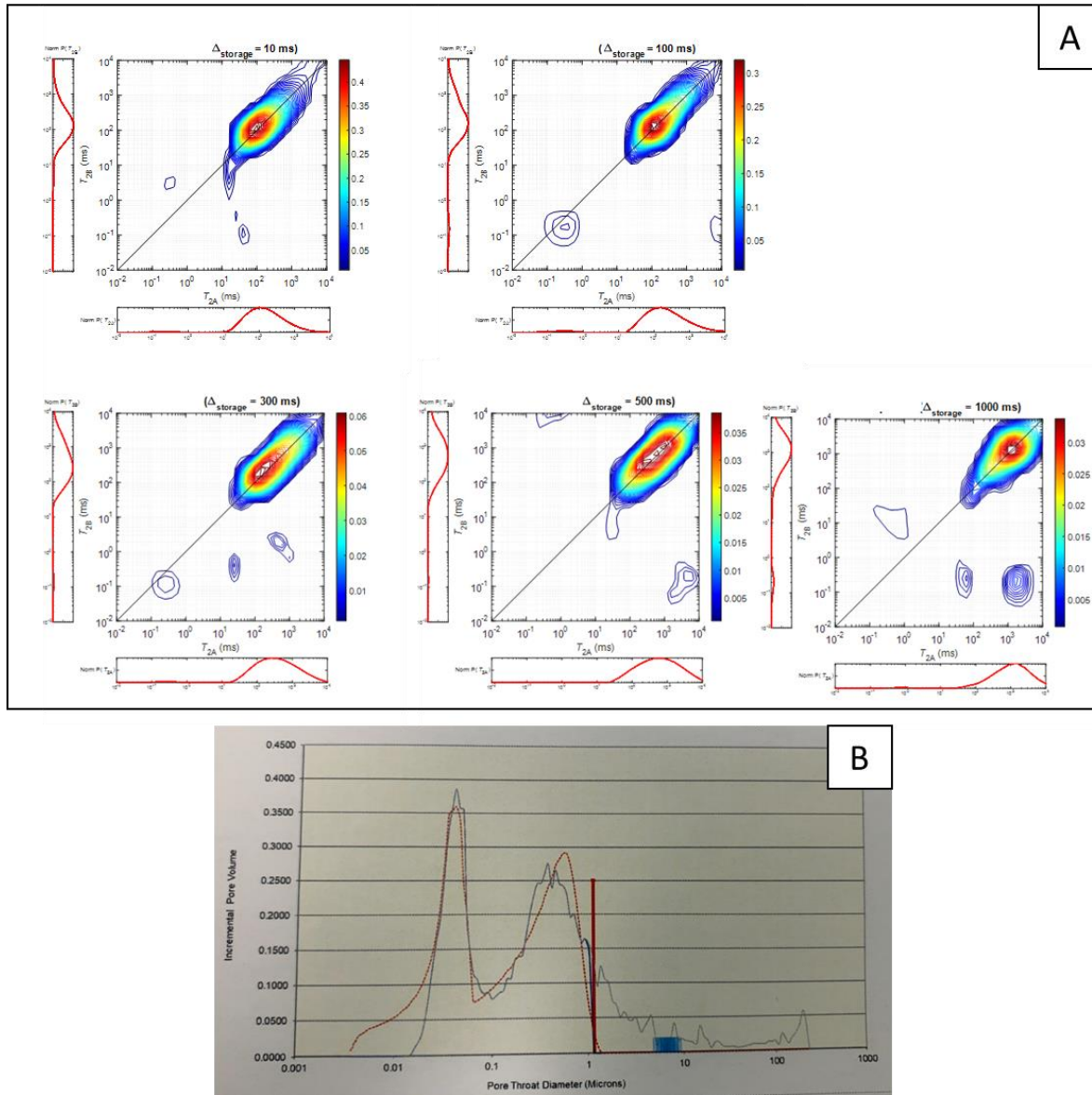


Figure 71: Sample 4 shows high degree of diffusion coupling by: A) 2D T2 shows a strong exchange between protons. Storage times used are 10, 100, 300, 500 and 1000 ms B) shows MICP analysis of sister sample indicating porosity bimodality, which was not captured i

Sample 6

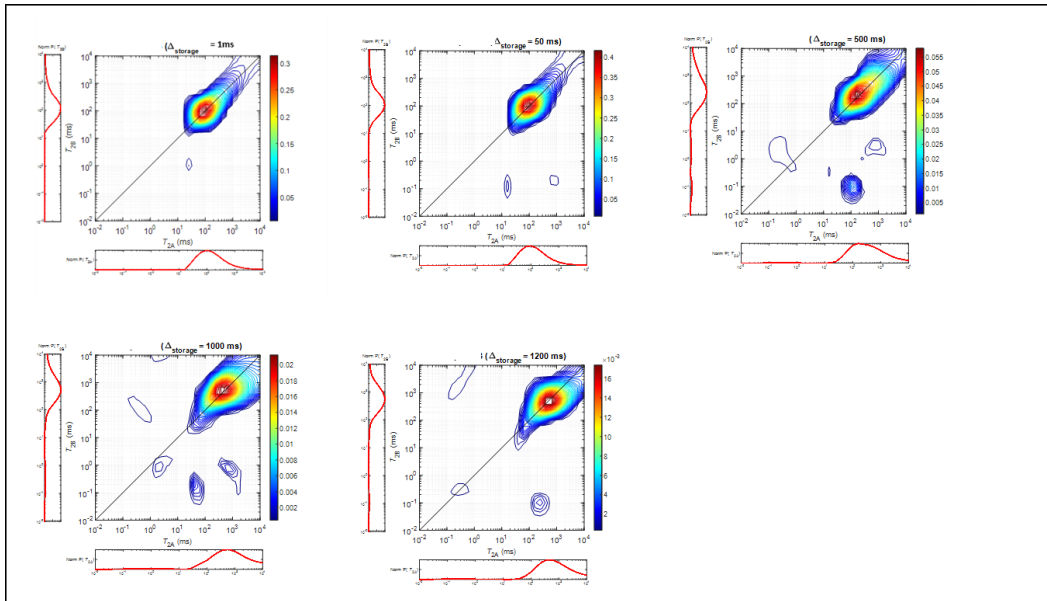


Figure 72: 2D T2 relaxation of core sample 6 showing relatively strong diffusion. The storage times used are: 1, 50, 500, 1000 and 1200 ms.

Sample 9

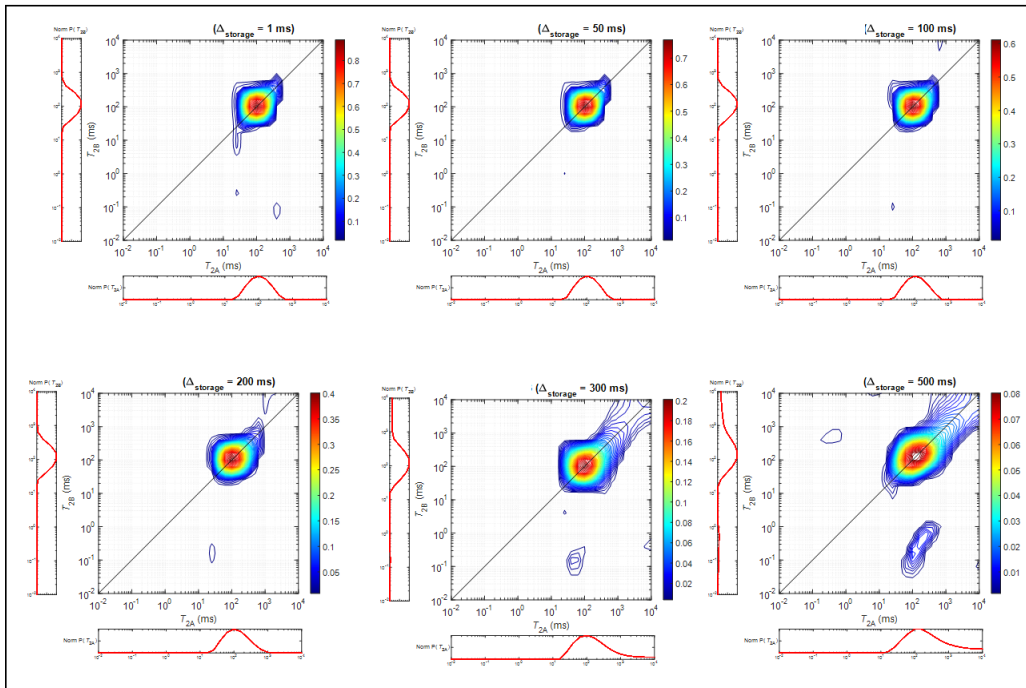


Figure 73: 2D T2 relaxation of core sample 9 showing intermedium diffusion. The storage times used are: 1, 50, 100, 200, 300 and 500 ms.

Sample 10

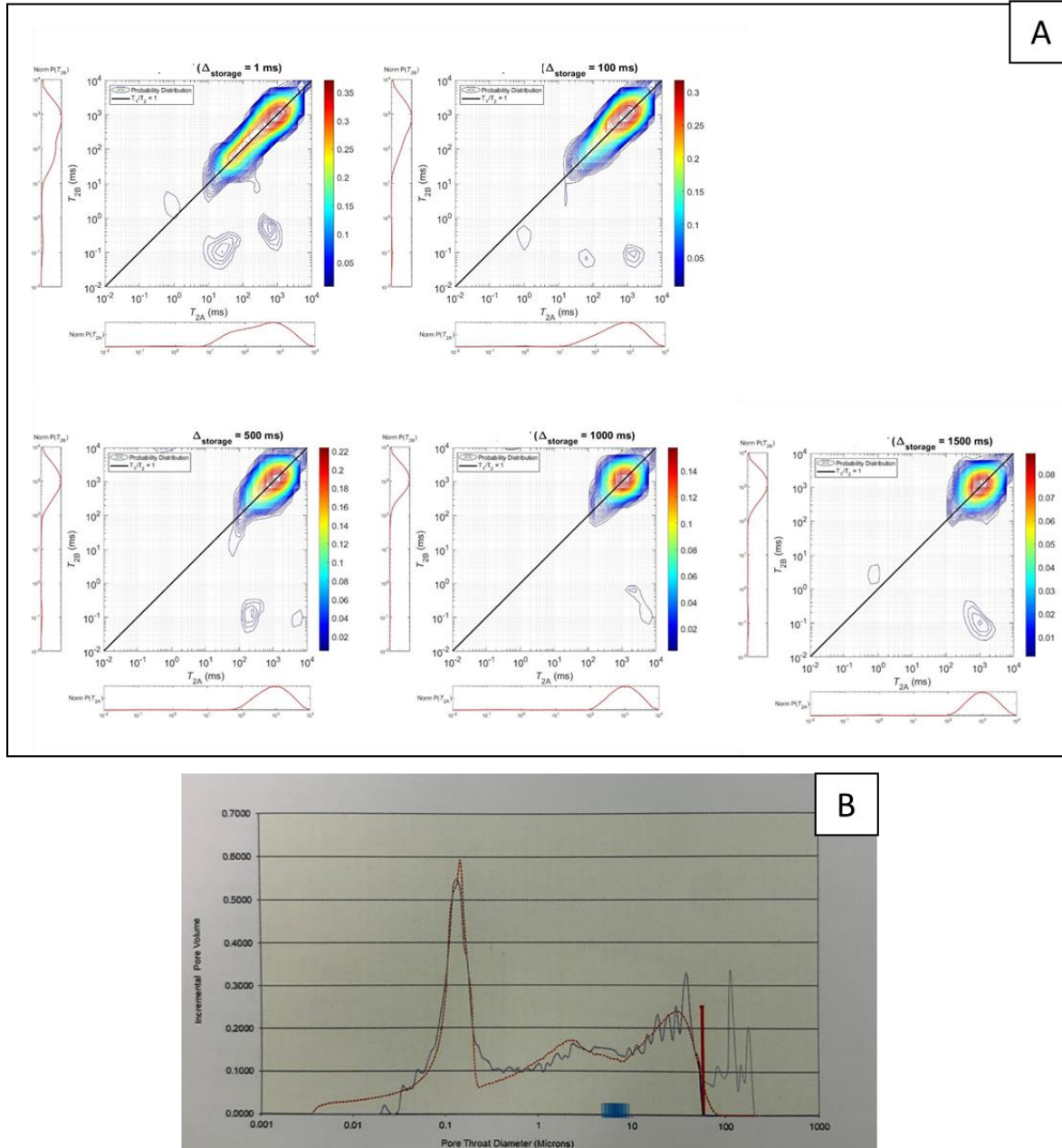


Figure 74: Sample 10 shows a high degree of diffusion coupling by: A) 2D T2 shows a strong exchange between protons. Storage times used are 1, 100, 1000 and 1500 ms B) shows MICP analysis of sister sample indicating porosity bimodality, which was not captured in t

Figure 75.A provides confirmation of the coupling status of sample 4. Draining the sample with incremental centrifugal speeds showed that water is leaving the macro spaces till reaching S_{wi} of 23.5% with a rotational speed of 12,000 RPM. The micro pore T_2 shows a

relaxation peak falling out of the range of the original relaxation of the fully saturated sample, indicating that the fully saturated relaxation is an average between micro and macro pore bodies, caused by diffusion coupling. Figure 75.B confirms from capillary pressure point of view, that the sample has reached S_{wi} . When introducing crude light oil to the sample, a clearer separation between the two peaks is established (Figure 76). Micro pore signal, which remains saturated with water, shows a consistent peak. The signal of the macro pore however, which has been saturated with Arab Light Crude oil, peaks at much higher amplitude and at slower time than the peak of water saturated curve. The oil signal is driven by the bulk relaxation of crude, as suggested by the clear proximity to the crude's bulk free relaxation peak. Aging the sample had a minimum effect on the relaxation, suggesting that there is no change in the wetting character of the sample, as shown in Figure 77. The reduction in micro pore amplitude could be attributed to the evaporation of water caused by aging.

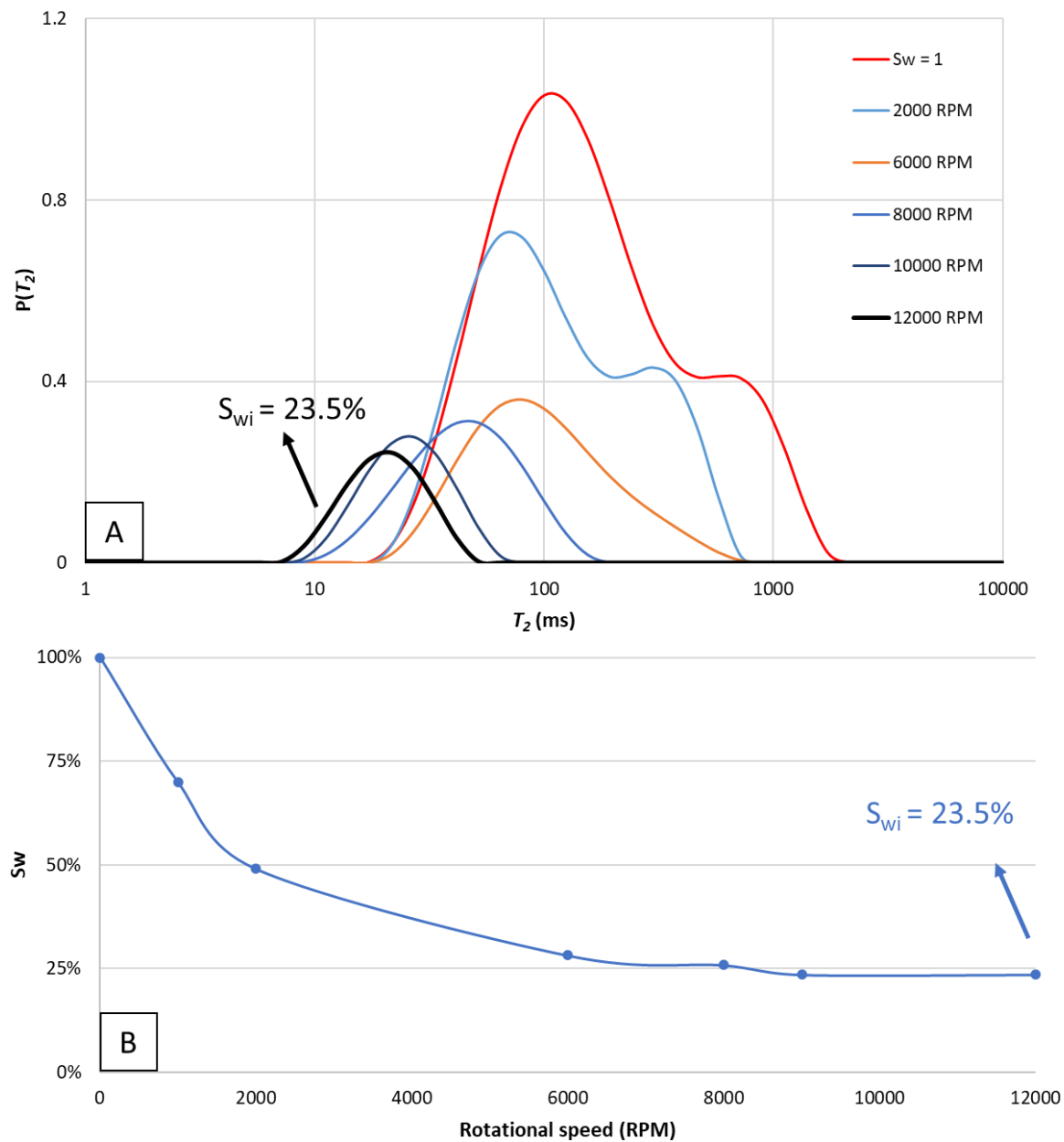


Figure 75: Showing the desaturation process of Sample 4 where: A) shows NMR T_2 relaxation after the completion of each centrifuge stage, till reaching $S_{wi} = 23.5\%$ at 12,000 RPM. B) shows the equilibrium of S_w with centrifuge.

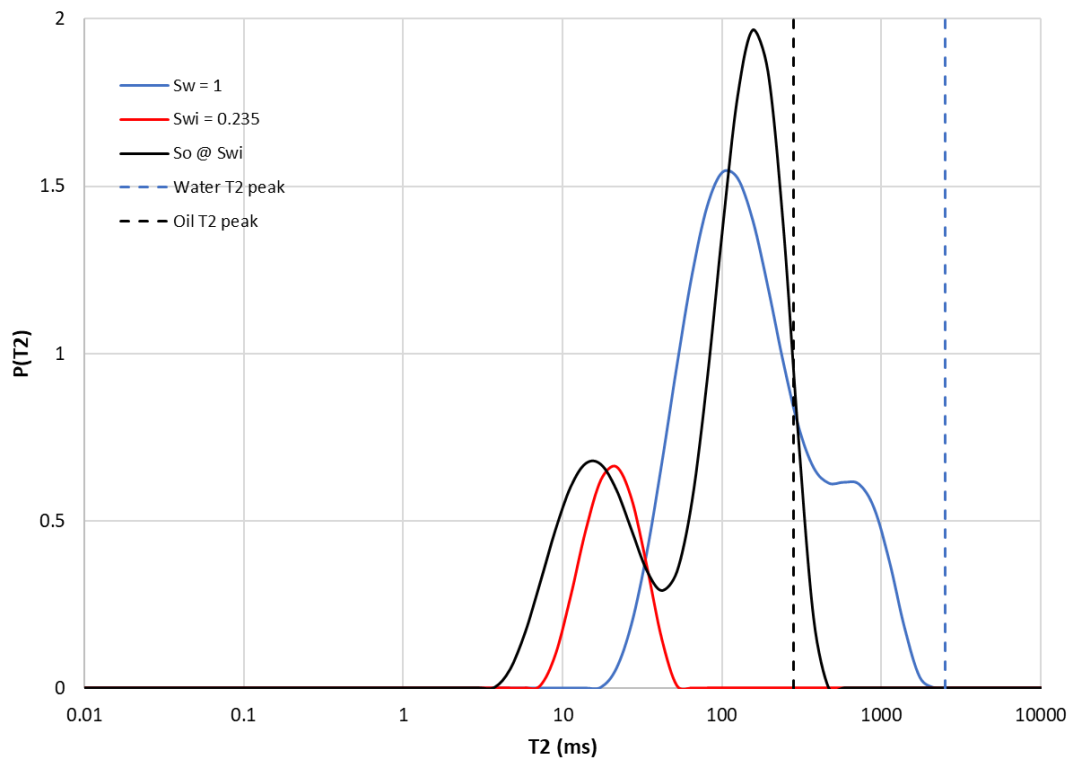


Figure 76: Comparison between Sample 4 T2 relaxation under different saturation endpoints; where blue solid line shows the relaxation when fully saturated with water, red curve shows the saturation when drained until reaching Swi and black solid line shows the re

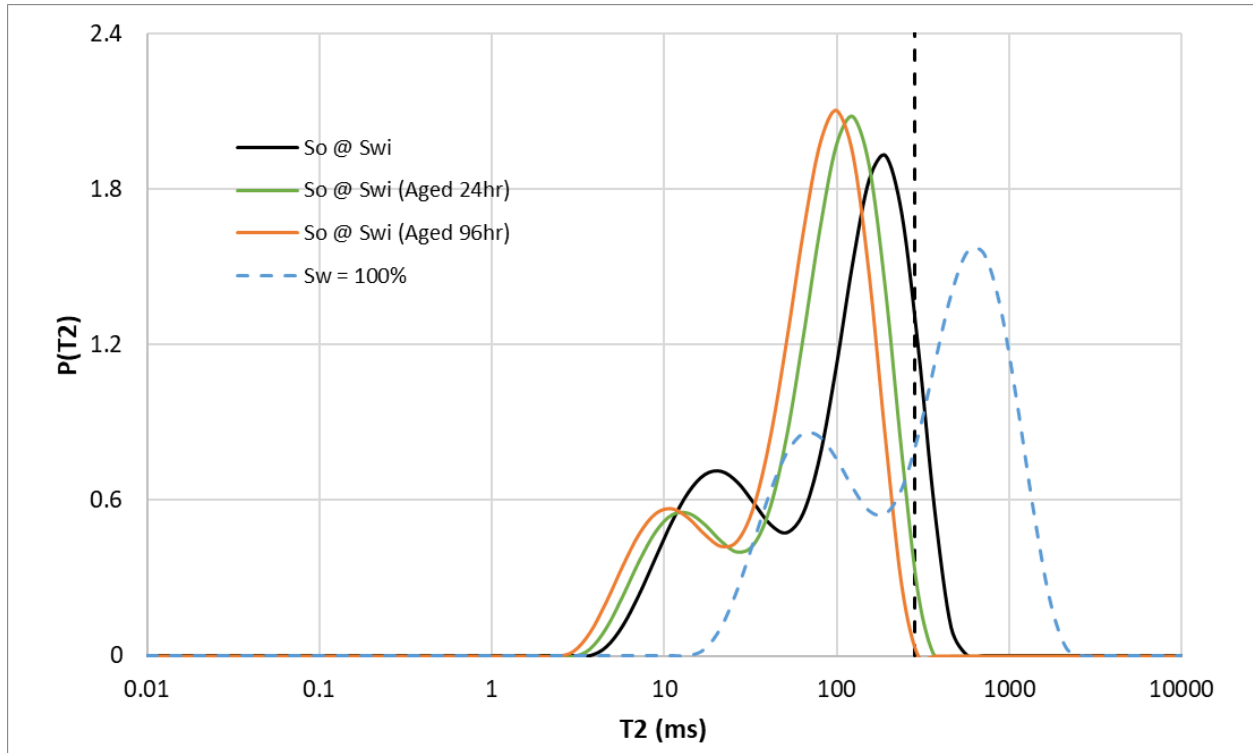


Figure 77: Sample 4 T2 relaxation after aging, showing consistent results between 1 and 4 days of aging.

For sample 2, it showed a similar trend to what was observed from sample 4. Figure 78 confirmed the diffusion coupling taking place in this sample, as the micro pore signal (achieved by centrifuge speed of 12,000 RPM), peaked at time lower than the relaxation time of the fully saturated (shown by the red curve). Going for higher centrifuge velocities might damage the sample integrity. However, this sample exhibited a much weaker diffusion strength compared to Sample 4. This is evident by the two interconnected distinct peaks from the relaxation of the fully saturated sample. Figure 79 shows the relaxation signal comparison between different saturation endpoints. Since the sample is weakly coupled, introducing oil to the fluid saturation system did not cause further separation. However, the peak of the larger pore shifted to the left, in consistency with the dominant fluid phase faster relaxation. The sample signal when saturated with oil at S_{wi} was dominated by the oil's bulk relaxation peak, where the amplitude of the macro system

increased at the expense of reduction in the micro pore system's peak. Similar to sample 2, Sample 2 aging did not result in any significant change, beside a slight budge to the left, as shown in Figure 80. All in all, despite 4 days of aging, the sample showed a dominant effect of fluid's bulk relaxation, suggesting that there's limited to no change in wettability character.

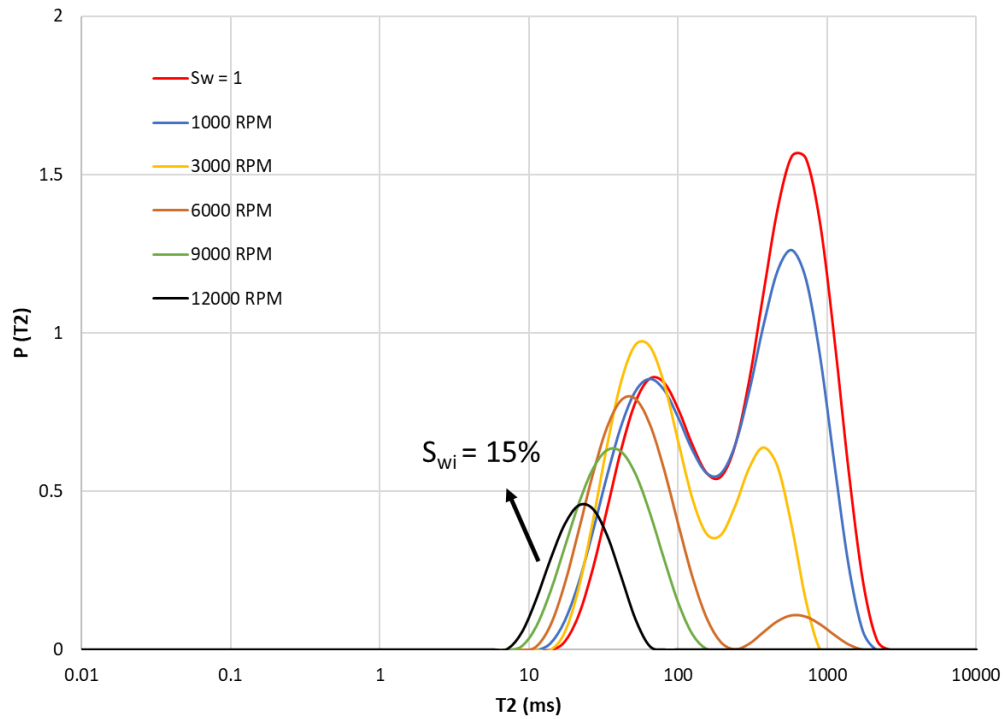


Figure 78: Showing the desaturation process of Sample 2 with NMR T2 relaxation signal measured after the completion of each centrifuge stage, till reaching $S_{wi} = 15\%$ at 12,000 RPM. B) shows the equilibrium of S_w with centrifuge.

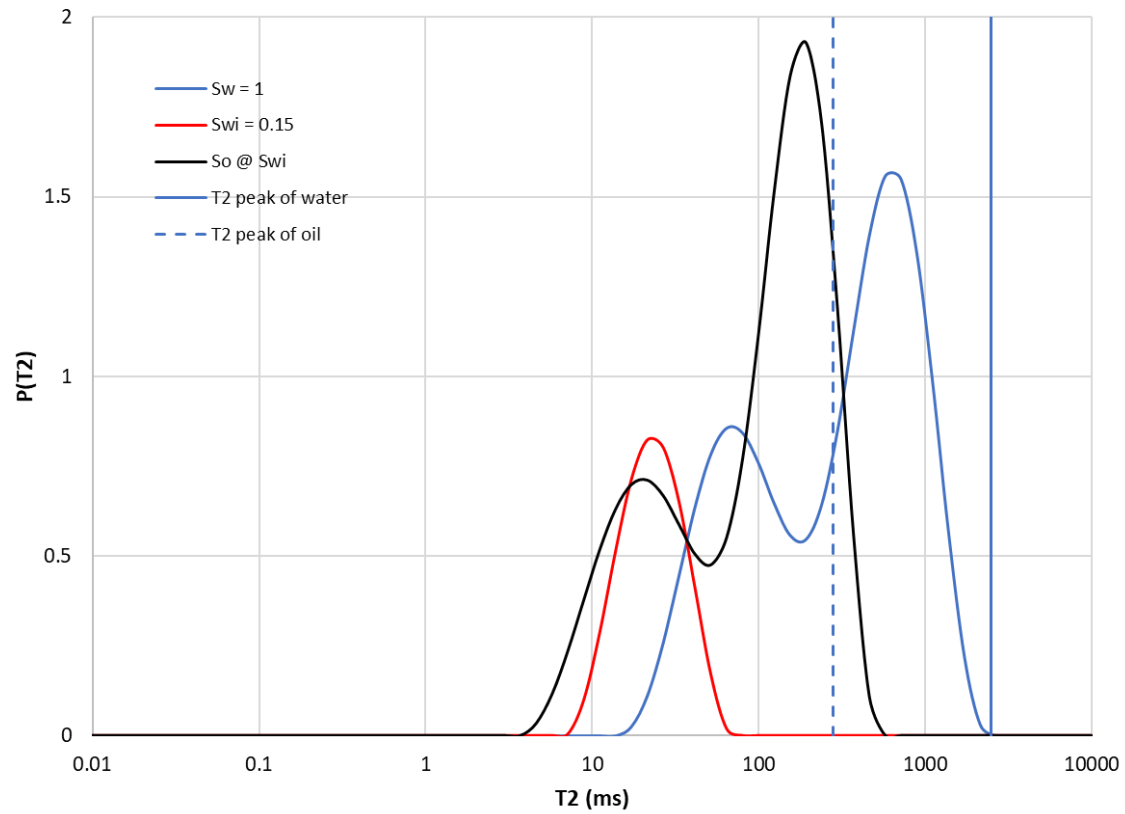


Figure 79: Comparison between Sample 2 T_2 relaxation under different saturation endpoints; where blue solid line shows the relaxation when fully saturated with water, red curve shows the relaxation when drained until reaching S_{wi} and black solid line shows the re

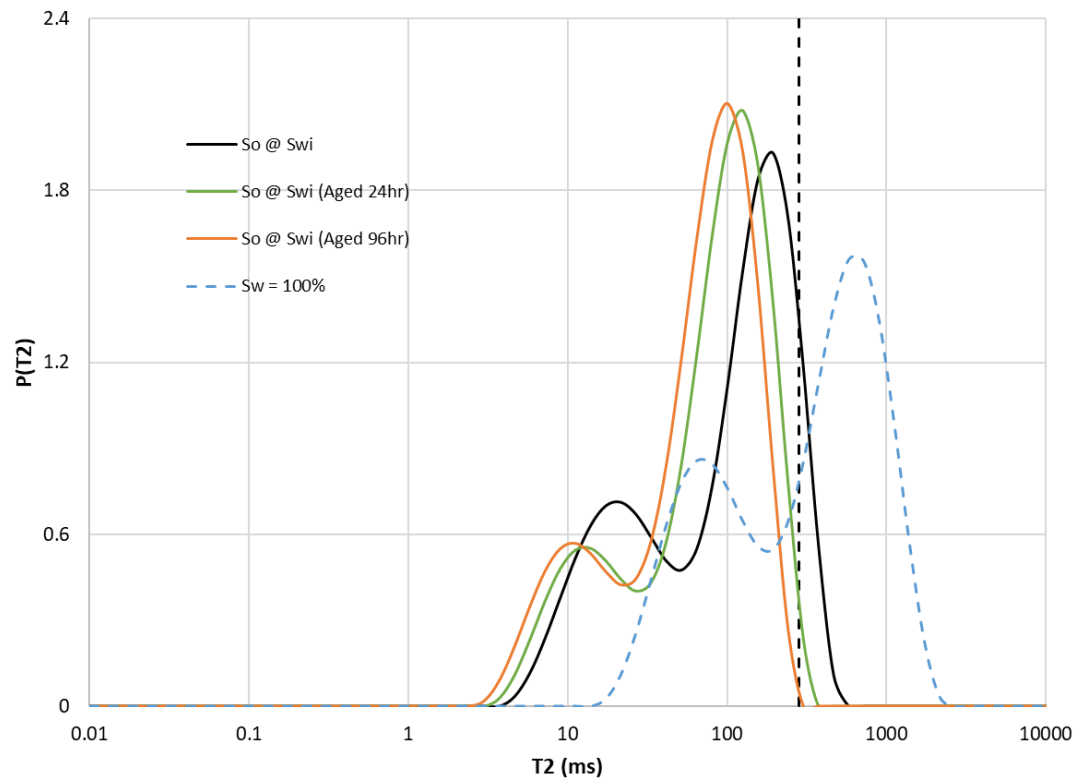


Figure 80: Sample 4 T2 relaxation after aging, showing consistent results between 1 and 4 days of aging.

CHAPTER 5

Conclusions

NMR offers unique petrophysical evaluation capabilities that positions it as one of the most important logging tools. With wide application portfolio spanning permeability estimation, fluid typing and saturation determination, and most importantly pore structure characterization. However, in complex, yet promising formations that have multiple porosity systems, NMR faces a challenge in interpretation caused by a phenomena called “Diffusion Coupling”. This originates when the relaxation of nuclear spins in a fluid is affected by the diffusion of the fluid molecules, leading to inaccurate estimation of the pore structure. When a fluid molecule diffuses from one pore to another, it can exchange magnetization with the fluid molecules in the new pore. This exchange of magnetization affects the relaxation of both exchanging pore bodies. This study investigated the diffusion coupling phenomena with a focus on the effects of fluid’s bulk relaxation and diffusion coefficients. As the diffusion coupling is affected by multiple interconnected factors, controlled environments (Sillica Gel samples and NMR Simulation) to investigate the phenomena, then validate the findings with field core plug samples.

The following conclusions were drawn from this work:

- Diffusion coupling is affected by the pore structure and distribution of porosity between the macro and micro. The plausibility of diffusion coupling occurrence increases in medium with lower contrast between macro and micro systems.

- Diffusion coupling strength increases with higher diffusion coefficient values in a nonlinear relationship.
- Fluid's free bulk relaxation affects coupling, owing to the wettability character. With a faster fluid free bulk relaxation, the diffusion coupling occurs with lower diffusion coefficient values.
- Fluid's diffusion coefficient and bulk relaxations are dependent on each other. Thus, any study should take both into considerations
- A sample exhibiting strong coupling under single phase saturation, can be decoupled when introducing another fluid with a big contrast between fluid's coefficients.

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